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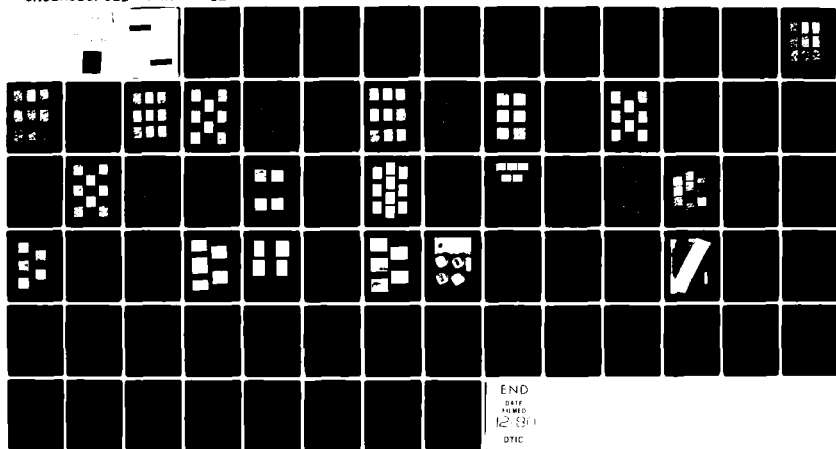
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WEAR RESISTANT COATINGS FOR TITANIUM.(U)  
JUL 80 P L MCDANIEL, R E FISHER

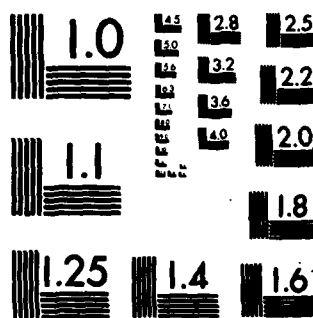
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| <p>During this third program phase, an intensive investigation was conducted to further evaluate the effect of various pulse settings on the properties of the coatings produced. Variations in cathode efficiency, molybdenum content, surface morphology, and hardness were related to changes in these pulse settings. The effects of plating solution composition and temperature on these properties were also studied. The completion of this third phase has provided additional information on the properties of chromium-molybdenum coatings on both steel and titanium, singled out specific pulse settings and heat treatments as optimal for producing wear resistant coatings on various alloys, and established standards for the properties and uses of the plating.</p> |  |   |      |

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## PREFACE

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## **SECTION I INTRODUCTION**

This report describes the results of the third year of a planned 4-year program to develop wear-resistant chromium-molybdenum coatings and to apply these coatings to candidate titanium alloy components. The capability to use titanium for gas turbine or airframe components often depends on a suitable surface treatment being available to alleviate inherent problems such as wear, erosion, and corrosion.

During the first year, it was established that a chromium-molybdenum coating can be applied to various titanium alloys as a solution to these problems. Also, because of the compatibility of the solid solutions formed by the chromium-molybdenum-titanium system, the effects of the coating process on the mechanical properties of titanium were minimal. In addition, it was demonstrated that plating with current interruption (pulse plating) can have a marked effect on the coating composition, increasing the molybdenum content.

In the second phase of the program, chromium-molybdenum coatings exhibited vastly superior wear resistance compared to conventional chromium plate. Pulse-plated deposits were found to have even better wear resistance than those produced with direct current on both titanium and steel. Various pulse times were investigated to determine their effect on the resulting coating. In addition, the chromium-molybdenum coating was found to have no significant effect on the tensile strength, yield strength, or elongation properties of titanium.

Although a reduction in the fatigue strength of one titanium alloy (fully heat-treated Ti 8Al 1Mo-1V) was noted, this was shown to be the result of the heat-treat process. Also, application of the coating increased the creep life up to 50% and protected titanium from oxidation at temperatures up to 760°C (1400°F). Finally, the coating process was successfully scaled up to plate titanium engine sync rings.

During this third program phase, an intensive investigation was undertaken to further evaluate the effect of various pulse settings on the properties of the coatings produced. Variations in cathode efficiency, molybdenum content, surface morphology, and hardness were related to changes in these pulse settings. The effects of plating solution composition and temperature on these properties were also investigated.

Also included in this phase was a study of the effect of various pulse settings on the wear resistance of chromium-molybdenum on both titanium and steel. Several heat treat cycles were evaluated for Ti 8Al-1Mo-1V and Ti 6Al-4V in an effort to maximize adhesion of the coating to the base metal. Further, the corrosion resistance afforded by the coatings was investigated by plating steel roller bearings at various pulse settings and subjecting them to salt-spray testing.

In addition, low-cycle fatigue tests were conducted on chromium-molybdenum coated Ti 8Al-1Mo-1V samples and fan blade roots were coated as a potential solution to galling problems.

As a culmination of the findings to date, a draft of a suggested Military Specification covering chromium-molybdenum coatings for both steel and titanium alloys was prepared and is submitted herein as Appendix D.

The completion of this third phase has:

- Provided additional information on the properties of chromium-molybdenum coatings on both titanium and steel
- Singled out specific pulse settings and heat treatments as optimal for producing wear resistant coatings on various alloys
- Established standards for the properties and uses of the plating.

## SECTION II EXPERIMENTAL PROGRAM

The experimental portion of this contract program contains the following:

1. Investigation of the effect of pulse-rectifier settings on the properties of chromium-molybdenum coatings
2. Evaluation of the effect of plating-bath composition and temperature on the deposition process
3. Determination of the effect of various pulse settings on the wear resistance of chromium-molybdenum on both titanium and steel
4. Evaluation of various diffusion heat-treat cycles for maximizing adhesion of the coating to a titanium substrate
5. Investigation to determine the corrosion resistance that the coating provided when plated onto bearing alloy steels
6. Evaluation of the effects of the coating process on low-cycle fatigue of Ti 8Al-1Mo-1V
7. Coating and testing of a NAVAIR-approved engine component
8. Providing a suggested Military Specification for chromium-molybdenum coatings.

The object of the third phase of this program was to evaluate the interrelationships of pulse setting, physical properties, and wear resistance.

### MATERIALS

Alloys used in this program are listed in Table 1 below:

TABLE 1. ALLOYS USED IN THIS INVESTIGATION

|                 | Type          | Test  |
|-----------------|---------------|---|
| Titanium Alloys | Ti 8Al-1Mo-1V | Pulse Effects<br>Wear (Pins)<br>Low-Cycle Fatigue<br>Blade Root Galling |
| Steel Alloys    | Ti 6Al-4V     | Wear (Pins)   |
|                 | SAE 3135      | Wear (Pins)   |
|                 | AISI 1137     | Wear (V-Blocks)   |
|                 | AMS 6490      | Corrosion   |

The chemicals used in the preparation of plating solutions and other solutions used in this investigation were all reagent-grade materials, except for the self-regulating chromic acid which was obtained from M&T Chemicals, Inc., under their designation SRHS CR 110.

## COATING APPLICATION PROCEDURE

Titanium specimens for pulse time and plating-bath composition and temperature studies were plated in accordance with the procedure detailed in Appendix A, except that no heat-treat was used. Titanium wear pins, low-cycle fatigue specimens, and fan blades were plated in the same manner with heat-treat included. The procedure outlined in Appendix B was employed to coat steel wear test pins and roller bearings.

## EFFECT OF PULSE SETTINGS

### On-Time Held at Three Milliseconds With Off-Time Varied

In this study, the off-time was increased while the on-time was held at 3 ms. This on-time was selected because the maximum Phase 2 molybdenum content was obtained using a 3 ms on-time and 3 ms off-time. Theoretically, the off-time is more critical than the on-time, because it determines whether the solution at the cathode reaches equilibrium before the next on-time. Figures 1 and 2 present scanning electron microscope (SEM) photos of the coatings before heat-treat, while Figure 3 details the cathode efficiencies and molybdenum content as a function of off-time.

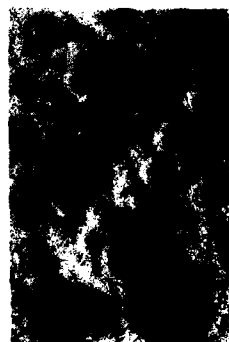
The pictures in Figures 1 and 2 show that no significant change in surface topography occurs until the off-time reaches 3 ms, although variations in grain size are present. There the coating takes on a plate-like appearance, as opposed to the nodular character of the previous samples. The molybdenum content shows little variance until the off-time reaches 2 ms, where it then peaks at 3 ms "off" and starts to decline until the next significant appearance change occurs at 20 ms. Those specimens with off-times of 20 and 30 ms have a needle-like appearance. Although they have a relatively high molybdenum content, the coating obtained was unsatisfactory. The samples appeared black, indicating that the Cr-Mo deposition mechanism has changed. This result may be partially explained by the concept of duty-cycle, which is defined as the on-time divided by the cycle time (on-time plus off-time). Most applications of pulse plating utilize duty cycles between 10 and 70%, as shown in Figure 3. In this case, on-times less than 1.3 ms or more than 27 ms are outside the recommended limits. Figure 3 also shows that an increase in molybdenum content is usually accompanied by a decrease in cathode efficiency and that the maximum molybdenum content is found at an off-time of 3 ms, confirming the results reported in Contract N00019-77-C-0330.

### Off-Time Held at Three Milliseconds With On-Time Varied

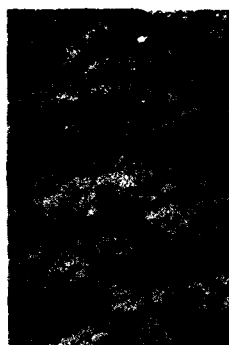
This study involved increasing the on-time, while holding the off-time constant at 3 ms. Figures 4 and 5 present SEM photos of this study. Figure 6 shows a plot of cathode efficiency and molybdenum content as a function of on-time.

Most of the SEM photos are similar to those produced in the previous study. A majority of the coatings produced at a duty cycle greater than 70% (on-time greater than 7 ms) have the same nodular topography as those produced in this duty cycle range in the previous study (off-time less than 1.3 ms). Variations in surface topography can be seen at 10 and 20 ms, where cigar-shaped grains of low molybdenum content are noted, and at 40 and 50 ms, where platelets are the predominant structure. The high molybdenum content (1.4%) at a 50 ms on-time seems suspect and may be in error since a sharp decrease in cathode efficiency is expected to accompany such a large increase in molybdenum content. For the samples produced within the recommended range, plate-like grains were the predominant structure, as in the preceding study. At a 7 ms on-time, the grains have a structure intermediate in appearance between the plates at a 5 ms on-time and the cigar-shaped particles at 10 ms "on." Also, the molybdenum content lies between those at these on-times; it is likely that this represents some type of transition structure.

(On-Time = 3 ms; Mag: 1000X)



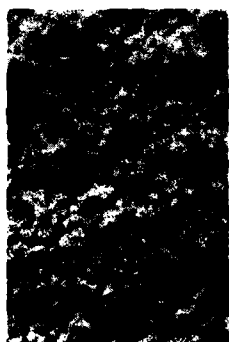
Off-Time = 0 ms (Direct)  
% CE = 24.0  
% Mo = 0.65



Off-Time = 0.1 ms  
% CE = 22.5  
% Mo = 0.65



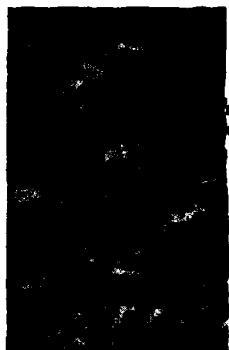
Off-Time = 0.2 ms  
% CE = 21.5  
% Mo = 0.70



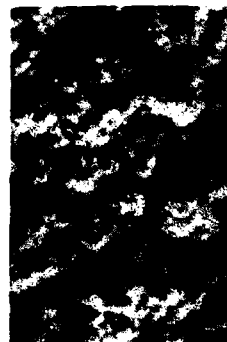
Off-Time = 0.3 ms  
% CE = 21.9  
% Mo = 0.85



Off-Time = 0.4 ms  
% CE = 20.9  
% Mo = 0.65



Off-Time = 0.5 ms  
% CE = 19.3  
% Mo = 0.70



Off-Time = 0.6 ms  
% CE = 19.0  
% Mo = 0.60



Off-Time = 0.7 ms  
% CE = 18.9  
% Mo = 0.70



Off-Time = 0.8 ms  
% CE = 18.4  
% Mo = 0.60

FD 151007

Figure 1. Specimens Pulse Plated in Unichrome-Based Solution, 0.0 to 0.8 ms Off-Time

( On Time = 3 ms; Mag: 1000X)



Off-Time = 0.9 ms  
% CE = 17.0  
% Mo = 0.60



Off-Time = 1.0 ms  
% CE = 17.1  
% Mo = 0.60



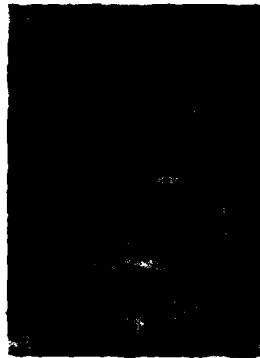
Off-Time = 2 ms  
% CE = 15.0  
% Mo = 1.0



Off-Time = 3 ms  
% CE = 13.6  
% Mo = 1.1



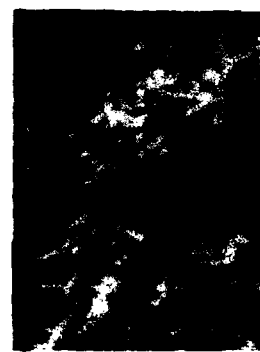
Off-Time = 5 ms  
% CE = 13.9  
% MO = 1.0



Off-Time = 7 ms  
% CE = 14.1  
% Mo = 0.8



Off-Time = 10 ms  
% CE = 15.9  
% Mo = 0.7



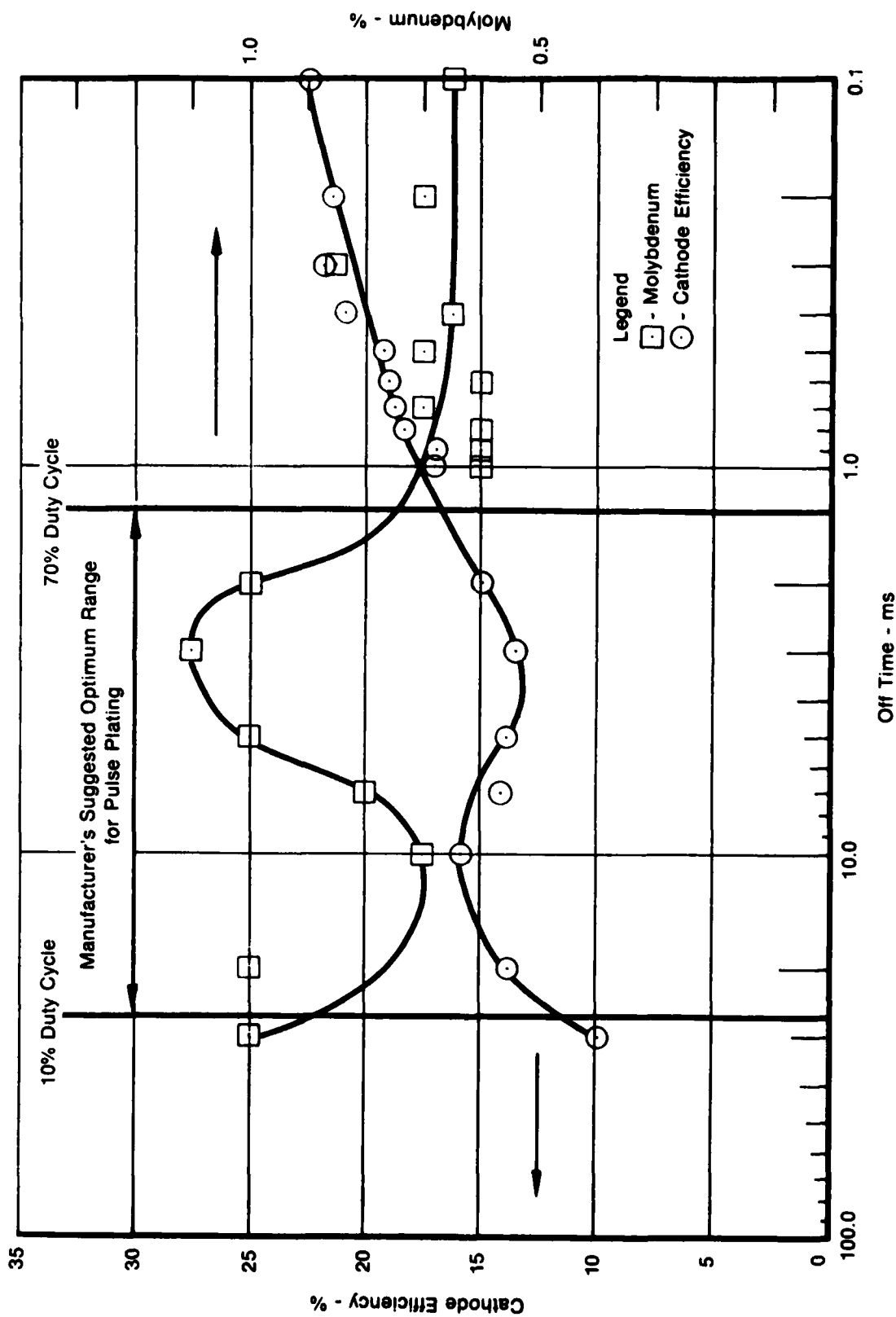
Off-Time = 20 ms  
% CE = 13.9  
% Mo = 1.0



Off-Time = 30 ms  
% CE = 10.0  
% Mo = 1.0

FD 151008

Figure 2. Specimens Pulse Plated in Unichrome-Based Solution, 0.9 to 30 ms Off-Time



FD 11-964

Figure 3. Influence of Off-Time on Molybdenum Content and Cathode Efficiency (On-Time = 3 ms)

(Off-Time = 3 ms; Mag: 1000X)



On-Time = 0.6 ms  
% CE = 17.1  
% Mo = 1.3



On-Time = 0.8 ms  
% CE = 15.1  
% Mo = 1.1



On-Time = 0.9 ms  
% CE = 16.0  
% Mo = 1.1



On-Time = 1.0 ms  
% CE = 14.7  
% Mo = 0.7



On-Time = 3.0 ms  
% CE = 15.4  
% Mo = 1.1



On-Time = 5.0 ms  
% CE = 14.5  
% Mo = 0.9



On-Time = 7.0 ms  
% CE = 16.6  
% Mo = 0.8



On-Time = 10.0 ms  
% CE = 20.4  
% Mo = 0.6



On-Time = 20.0 ms  
% CE = 16.9  
% Mo = 0.6

FD 152000

Figure 4. Specimens Pulse Plated in Unichrome-Based Solution, 0.6 to 20 ms On-Time



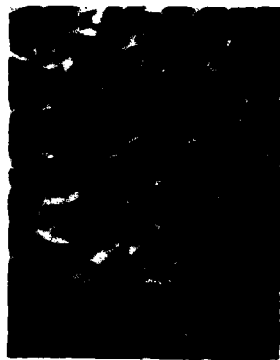
(Off-Time = 3 ms; Mag: 1000X)



On-Time = 30 ms  
% CE = 16.4  
% Mo = 0.7



On-Time = 40 ms  
% CE = 16.6  
% Mo = 0.65



On-Time = 50 ms  
% CE = 18.2  
% Mo = 1.4



On-Time = 60 ms  
% CE = 18.1  
% Mo = 0.9



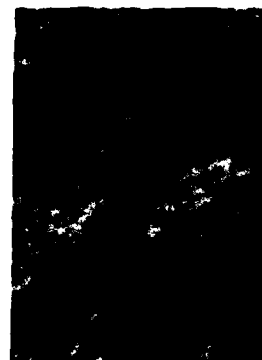
On-Time = 70 ms  
% CE = 22.4  
% Mo = 0.7



On-Time = 80 ms  
% CE = 18.3  
% Mo = 0.8



On-Time = 90 ms  
% CE = 19.6  
% Mo = 0.6



On-Time = 99.9 ms  
% CE = 19.8  
% Mo = 0.7

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Figure 5. Specimens Pulse Plated in Unichrome-Based Solution, 30 to 99.9 ms On-Time

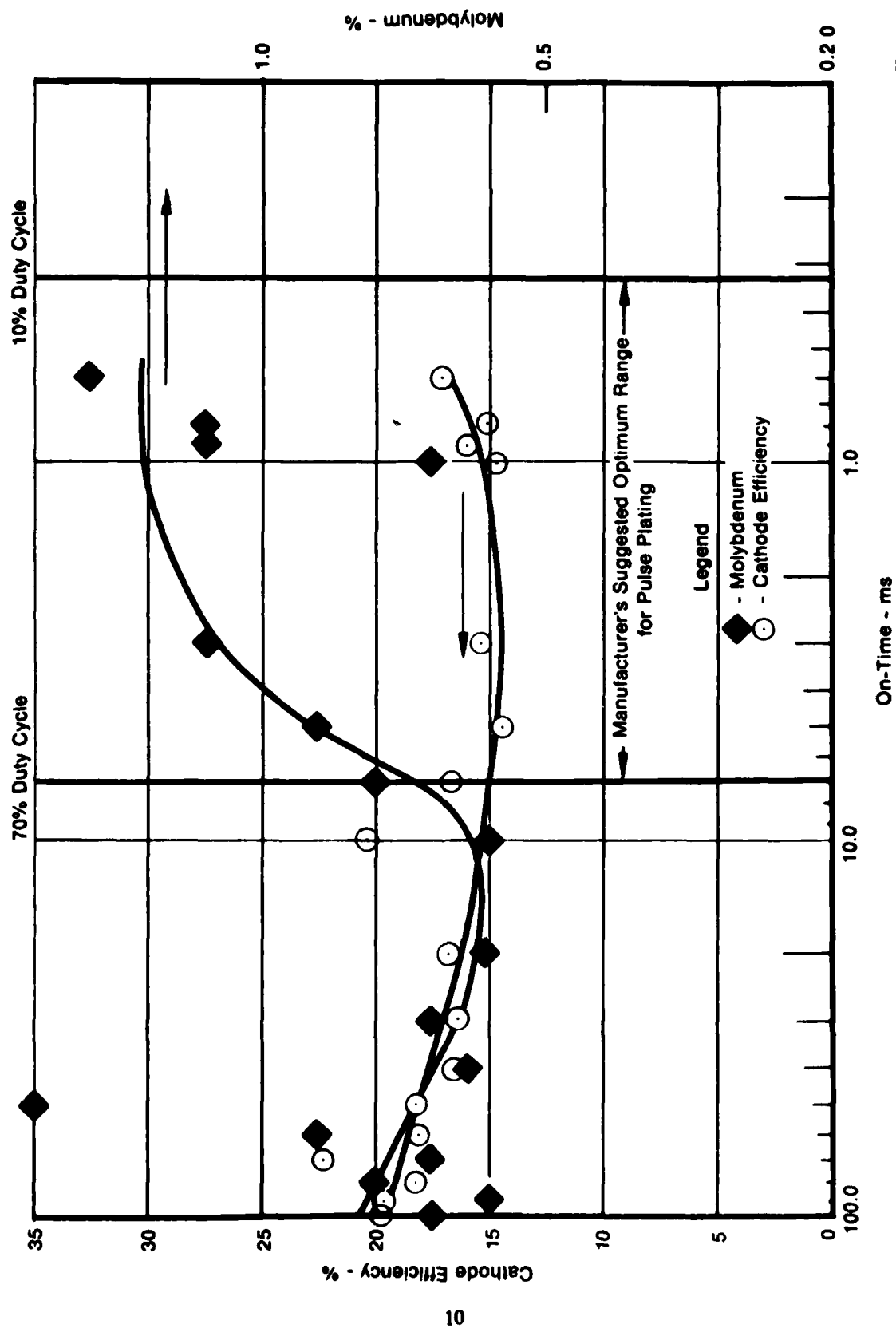


Figure 6. Influence of On-Time on Molybdenum Content and Cathode Efficiency (Off-Time = 3 ms)

The cathode efficiency and molybdenum content increase, while the average size of the plates decreases as the on-time approaches zero. Figure 6 does not show the same inverse relationship between changes in cathode efficiency and in molybdenum content seen above. The cathode efficiencies do not vary much with on-time. This bears out the theory that varying the off-time is more critical than varying the on-time.

#### **On-Time Held At 99.9 Milliseconds With Off-Time Varied**

In this study, the off-time was increased while the on-time was held constant at 99.9 ms. Figure 7 presents the SEM photos for this study and Figure 8 presents a plot of cathode efficiency and molybdenum content as a function of off-time.

Most of the SEM photos show a nodular surface topography, as has been the case with most samples produced with a duty cycle greater than 50%. At an off-time of 89.9 ms, tiny rice-shaped grains are interspersed with the nodules and at an off-time of 99.9 ms these grains have become the predominant structure, closely packed together to form a deposit with a good physical appearance, high molybdenum content (1.2%), and a cathode efficiency (23.1%) almost as high as that obtained with direct plating. Figure 8 shows that the cathode efficiency increases with off-time up to 99.9 ms, while the molybdenum content decreases slightly until the structural change occurs.

#### **Off-Time Held at 99.9 Milliseconds With On-Time Varied**

This study involved holding the off-time at 99.9 ms while varying the on-time. Figure 9 presents the SEM photos of the study and Figure 10 presents a plot of cathode efficiency and molybdenum content as a function of on-time.

SEM photos show that two basic structures were formed: (1) fine, oblong grains and (2) square platelets of relatively large size. As the on-time decreased, the structure began to change and the molybdenum content decreased. At an on-time of 89.9 ms, the rice-shaped grains mentioned previously became larger, and, when the on-time was decreased to 79.9 ms, square plates began to replace the grains. At an on-time of 69.9 ms, these plates became the dominant structure. Although the molybdenum content at 59.9 and 49.9 ms "on" is quite high, the blackened appearance of the coatings indicates that the Cr-Mo deposition process changed. Figure 10 shows that the molybdenum content levels out at on-times greater than 69.9 ms, once again indicating the relative importance of off-time.

#### **Evaluating Overall Trends**

To facilitate the evaluation of the results obtained in this program, eight additional samples were plated at random on- and off-times to fill in gaps in the existing data. SEM photos and the essential information about each setting chosen are shown in Figure 11. All but one of the samples show a high molybdenum content, and most of these exhibit an extremely small grain size. The samples plated at 60 ms "on", 20 ms "off" and at 80 ms "on", 60 ms "off" have grains shaped like rectangular blocks, but still show a high molybdenum content. The only sample with low molybdenum content (0.65%) was plated at 90 ms "on", 20 ms "off", and the coating has an appearance much like direct-plated Cr-Mo. The cathode efficiencies are relatively low, ranging from 8.6 to 14.5%.

Figure 12 presents a plot of cathode efficiency versus the percent duty cycle. For a setting with a duty cycle greater than 70%, a cathode efficiency of greater than 16% will probably result. A curve is drawn through the points representing settings with 3 ms on-time. Cathode efficiency tends to increase with percent duty cycle. The data points produced with a constant off-time of 3 ms do not fit this curve, but show the same general trend. The points representing pulse cycles

( On Time = 99.9 ms; Mag: 1000X)



Off-Time = 3 ms  
% CE = 19.8  
% Mo = 0.7



Off-Time = 19.9 ms  
% CE = 19.1  
% Mo = 0.7



Off-Time = 29.9 ms  
% CE = 19.5  
% Mo = 0.7



Off-Time = 39.9 ms  
% CE = 20.6  
% Mo = 1.6



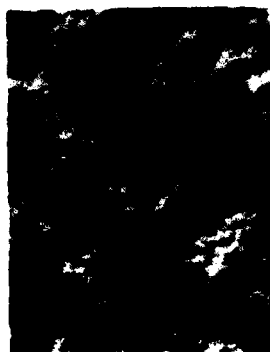
Off-Time = 49.9 ms  
% CE = 22.1  
% Mo = 0.8



Off-Time = 69.9 ms  
% CE = 21.8  
% Mo = 0.5



Off-Time = 79.9 ms  
% CE = 22.6  
% Mo = 0.65



Off-Time = 89.9 ms  
% CE = 23.0  
% Mo = 0.6



Off-time = 99 ms  
% CE = 23.1  
% Mo = 0.7

FD 144103

Figure 7. Specimens Pulse Plated in Unichrome-Based Solution, 3.0 to 99.9 ms Off-Time

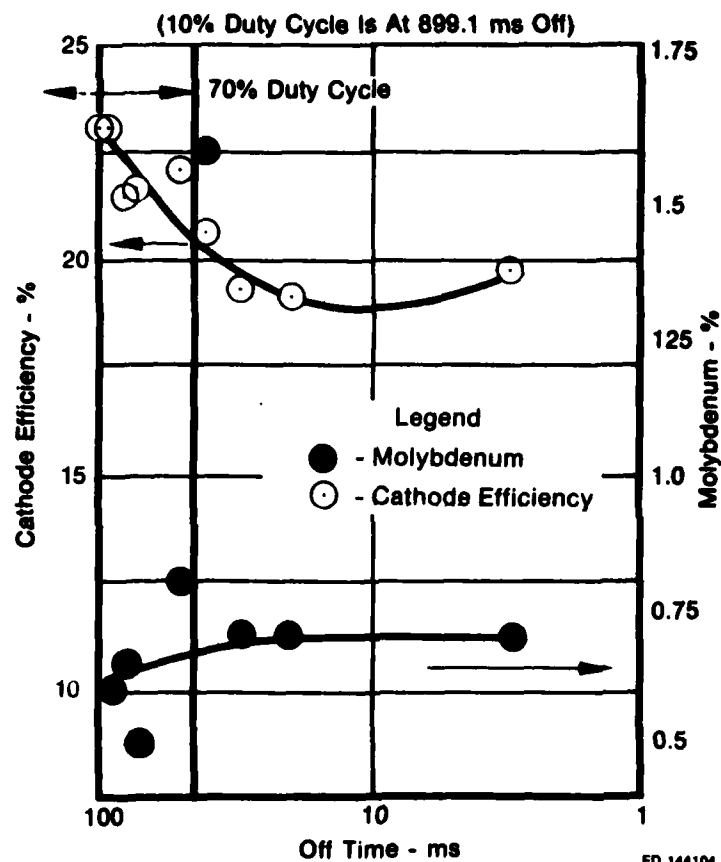
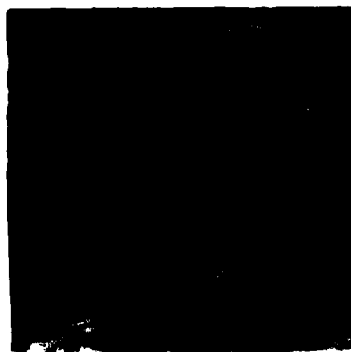


Figure 8. Influence of Off-Time on Molybdenum Content and Cathode Efficiency (On-Time = 99.9 ms)

(Off Time = 99.9 ms; Mag: 1000X)



On-time = 99.9 ms  
% CE = 23.1  
% Mo = 0.7



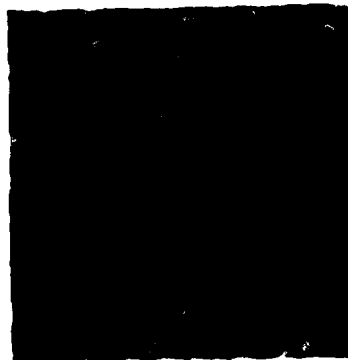
On-Time = 89.9 ms  
% CE = 20.9  
% Mo = 0.7



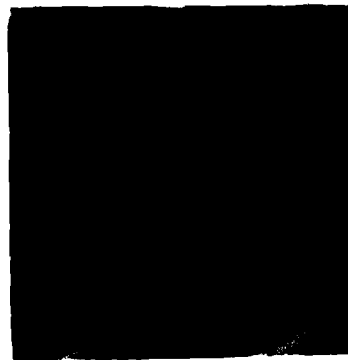
On-Time = 79.9 ms  
% CE = 21.4  
% Mo = 0.7



On-Time = 69.9 ms  
% CE = 23.1  
% Mo = 0.7



On-Time = 59.9 ms  
% CE = 21.9  
% Mo = 1.5



On-Time = 49.9 ms  
% CE = 17.0  
% Mo = 1.3

FD 144105

Figure 9. Specimens Pulse Plated in Unichrome-Based Solution, 99.9 to 49.9 ms On-Time

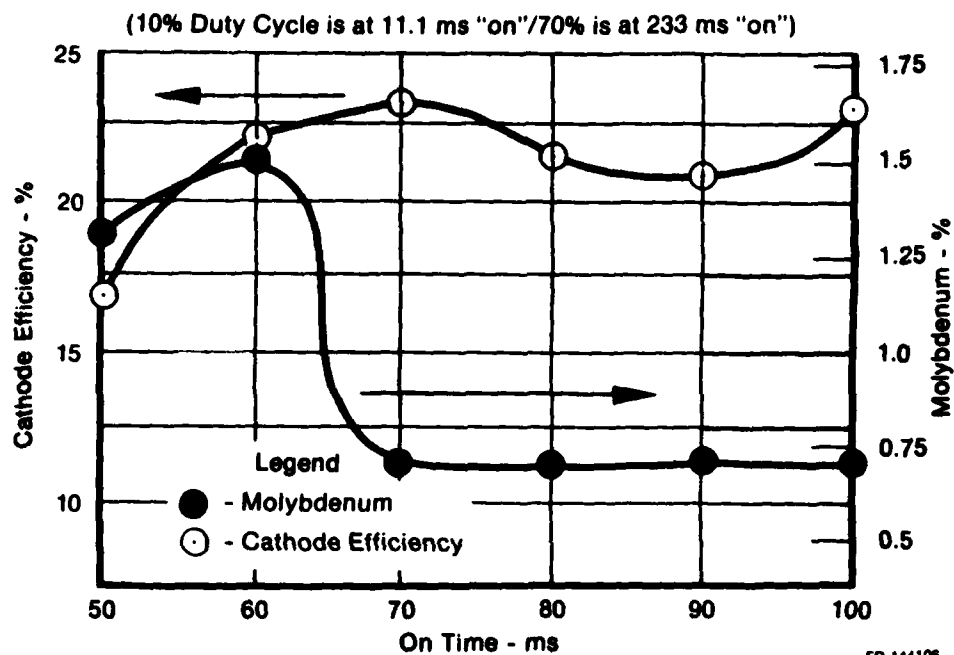


Figure 10. Influence of On-Time on Molybdenum Content and Cathode Efficiency (Off-Time = 99.9 ms)

(Mag: 1000X)

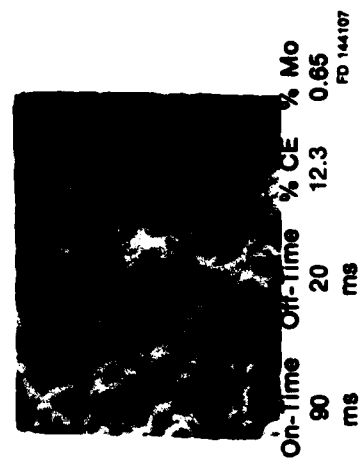
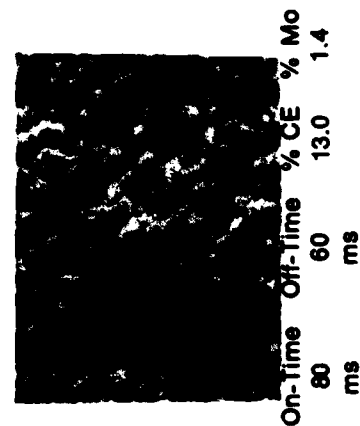
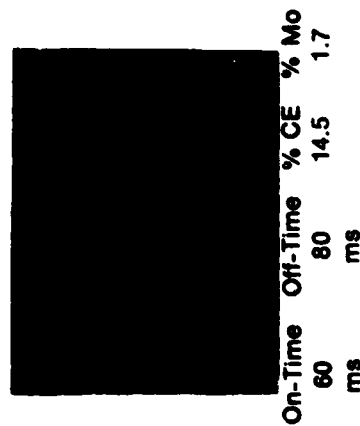
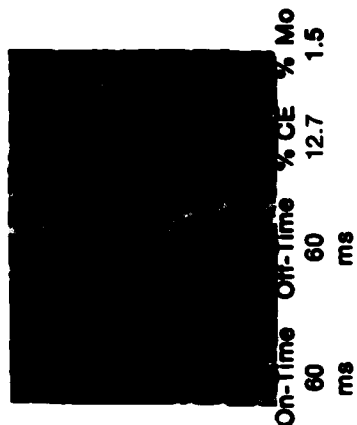
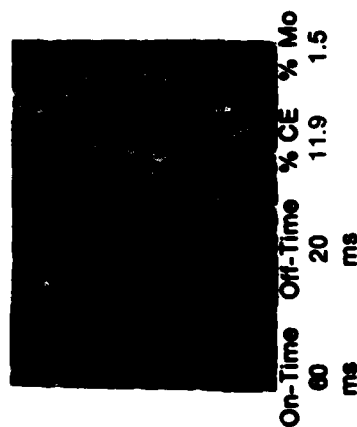
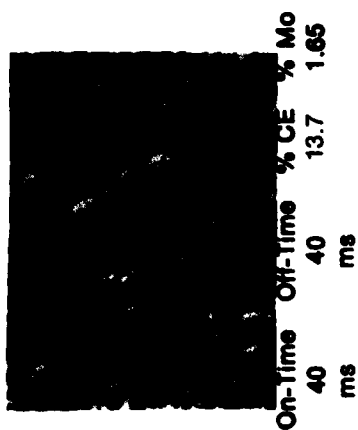
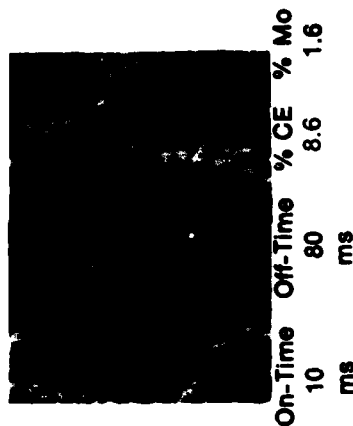
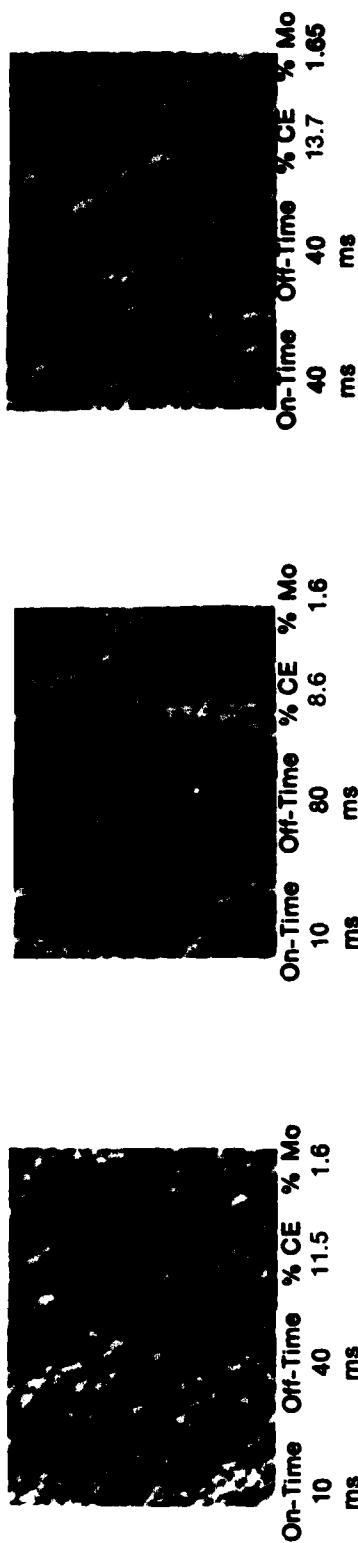


Figure 11. Specimens Pulse Plated in Unichrome-Based Solution, Random On/Off-Times



with "on" or "off" times of 99.9 ms show a decrease in cathode efficiency with increasing duty cycle. These points have a separate curve drawn through them. An attempt was made to plot the molybdenum content against the percent duty cycle, but the data was so scattered that no trends could be developed. However, virtually all settings with a duty cycle greater than 70% resulted in a coating with molybdenum content of less than 0.85%.

Figure 13 develops a plot of cathode efficiency versus off-time with curves drawn in for selected on-times. Only on-times with at least three data points were plotted. The change in form between on-times of 10 ms and 60 ms indicates that a change in the deposition mechanism takes place at some point between these two values.

Another characteristic that was evaluated is the hardness of the coating. Figure 14 shows the results of the evaluation. For a given on-time, hardness decreases with increasing off-time. Further, the rate of this decrease becomes less pronounced as the on-time is increased. All samples plated with an off-time of 99.9 ms showed a Vickers hardness number between 210 and 240, suggesting that hardness values approach a value independent of on-time as off-time is increased. This independence serves to reinforce the theory that off-time is more important than on-time.

Attempts to determine the density of the coatings met with limited success. Several materials were plated with a thick Cr-Mo coating, including aluminum and copper, but attempts to separate the base material and the coating proved unsuccessful. Many of the titanium samples were tested for specific gravity before and after plating, but few of the density results obtained seemed reasonable. A Cr-Mo coating containing 2% molybdenum would have a maximum possible density of 7.26 g/cm<sup>3</sup>, based on the densities of the pure materials. For the twelve densities obtained with values less than this, the average was 5.05 g/cm<sup>3</sup>.

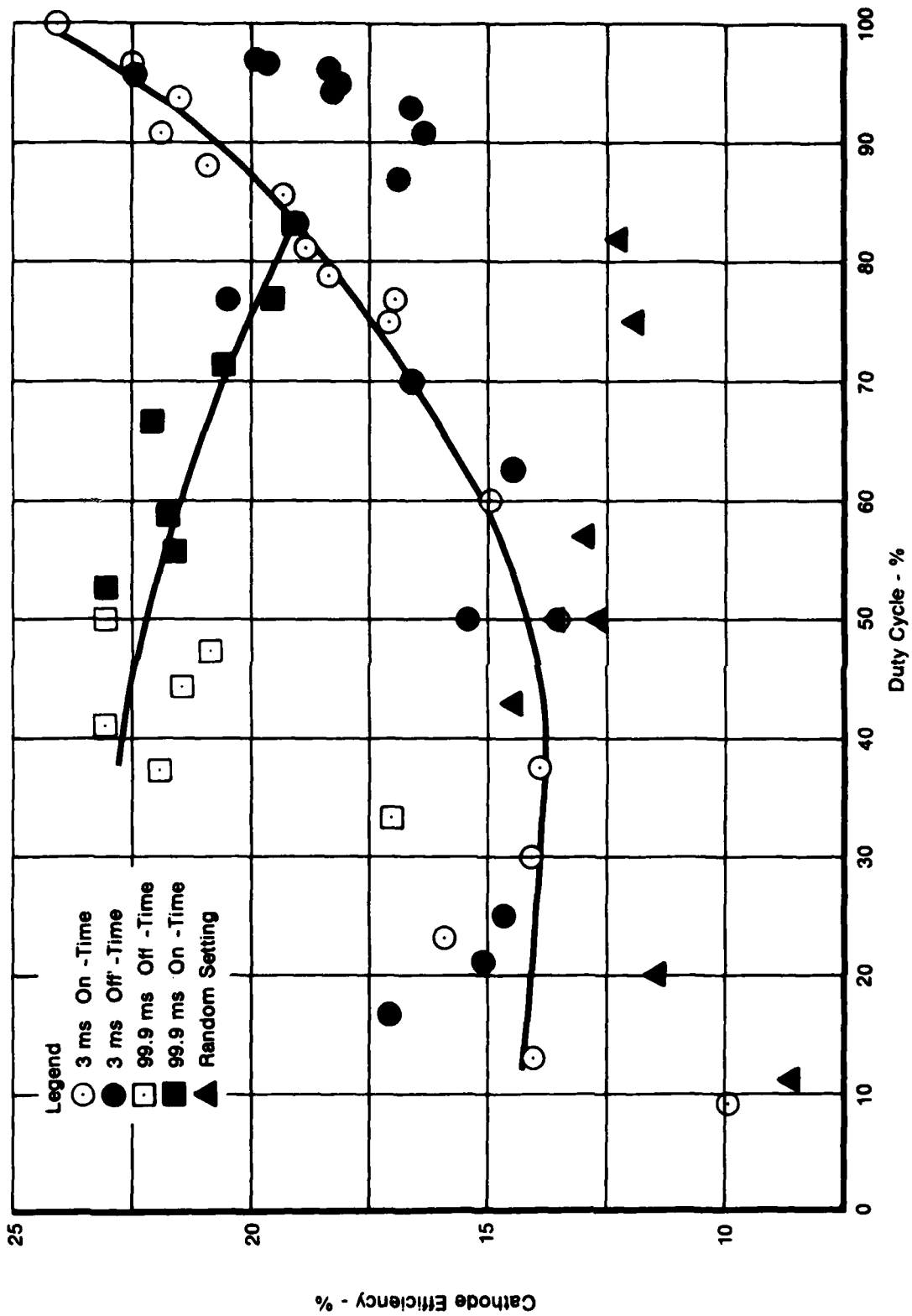
## EFFECT OF PLATING BATH COMPOSITION AND TEMPERATURE

### Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>) Concentration

The plating solution used in this study was prepared with chromium trioxide, rather than self-regulating Unichrome, which contains a predetermined amount of sulfate. The use of chromium trioxide (CrO<sub>3</sub>) permits the control of the sulfate content of the solution by the addition of discrete amounts of sulfuric acid. Appendix C describes the solution preparation. Samples were vapor blasted and etched as before.

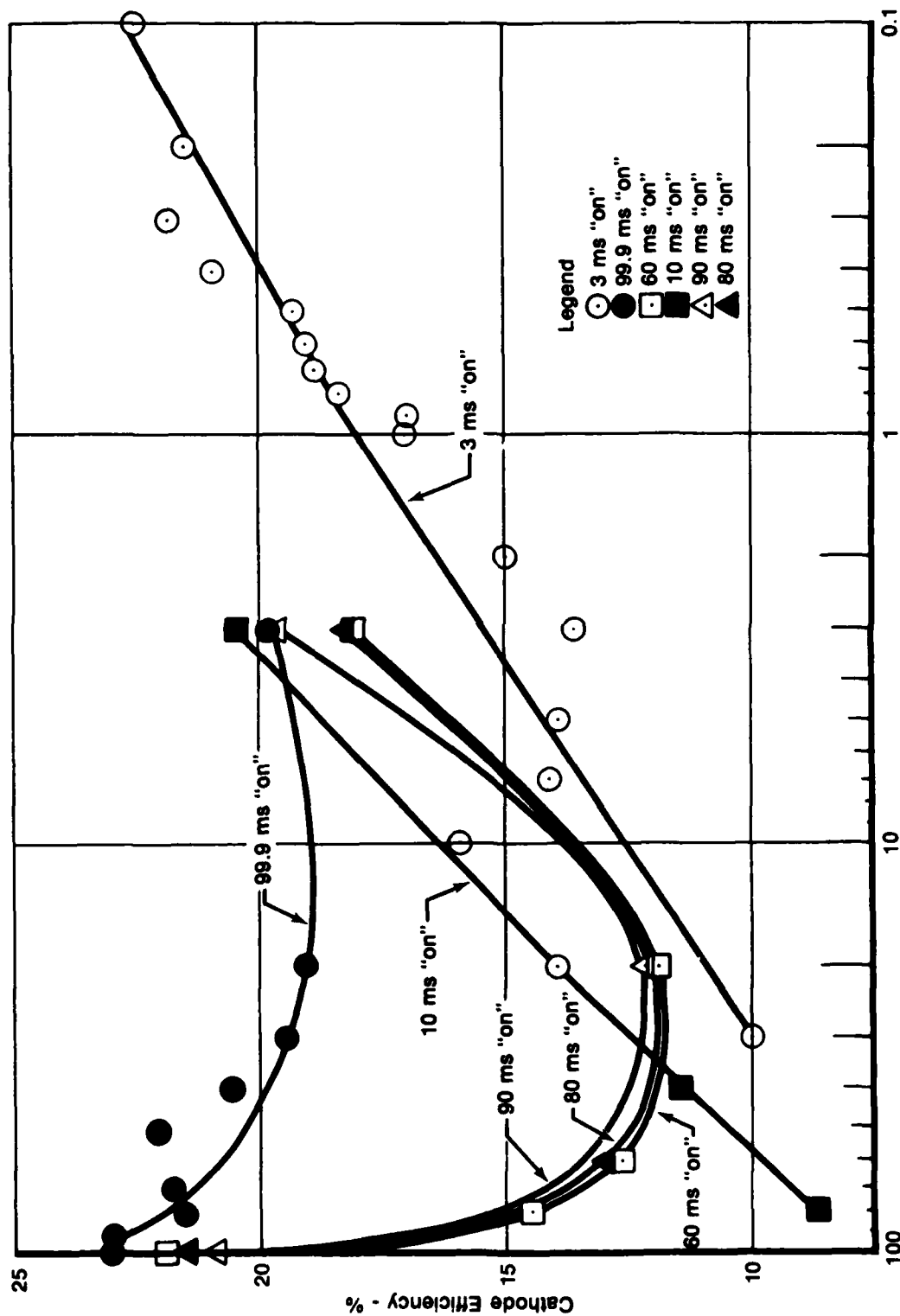
A pulse time of 40 ms "on", 40 ms "off" was selected for this study because it resulted in a cathode efficiency almost identical to that observed at 3 ms "on", 3 ms "off" (the optimum on/off time from Phase 2), yet the molybdenum content was 50% higher (1.65%). Figure 15 shows the results through SEM photos and Figure 16 presents a plot of cathode efficiency and molybdenum content against H<sub>2</sub>SO<sub>4</sub> concentration.

SEM photos reveal that at H<sub>2</sub>SO<sub>4</sub> concentrations of 0.1, 0.2, and 0.3 milliliters per liter (ml/l) of plating solution, no deposition occurs and a cathode efficiency of zero was obtained. At 0.5 ml/l, a slight fine nodular coating of blackened appearance containing 0.34% molybdenum was deposited with very low cathode efficiency. When the H<sub>2</sub>SO<sub>4</sub> concentration was increased to 1.0 ml/l, a coating consisting of closely-packed, randomly-shaped platelets resulted. The cathode efficiency increased to 18.9% and molybdenum content rose to 0.72%. A further increase in H<sub>2</sub>SO<sub>4</sub> concentration to 1.5 ml/l resulted in a decrease in both the size of the platelets and the molybdenum content, but the cathode efficiency rose to 31.0%.



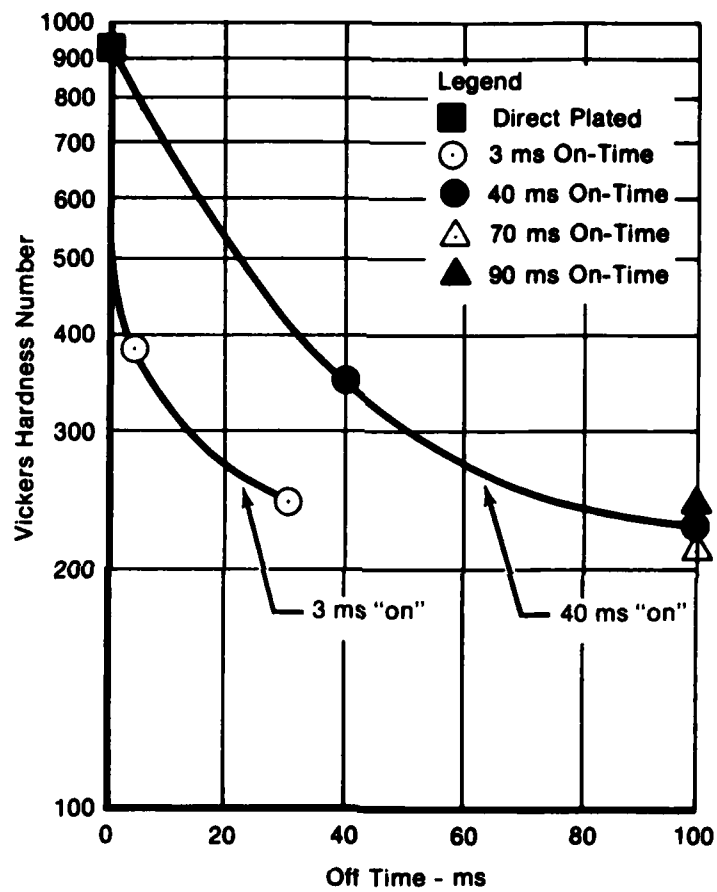
FD 144108

Figure 12. Cathode Efficiency versus Percent Duty Cycle



FD 144109

Figure 13. Cathode Efficiency vs Off-Time for Selected On-Times



FD 144110

Figure 14. Hardness vs Off-Time (Before Heat Treat)

(On - Off Time = 40 ms; Mag: 1000X)



ml  $\text{H}_2\text{SO}_4/\text{l} = 0.1$   
% CE = 0  
% Mo = 0.1



ml  $\text{H}_2\text{SO}_4/\text{l} = 0.2$   
% CE = 0  
% Mo = 0.11



ml  $\text{H}_2\text{SO}_4/\text{l} = 0.3$   
% CE = 0  
% Mo = 0.13



ml  $\text{H}_2\text{SO}_4/\text{l} = 0.5$   
% CE = 1.2  
% Mo = 0.34



ml  $\text{H}_2\text{SO}_4/\text{l} = 1.0$   
% CE = 18.9  
% Mo = 0.72



ml  $\text{H}_2\text{SO}_4/\text{l} = 1.5$   
% CE = 31.0  
% Mo = 0.39



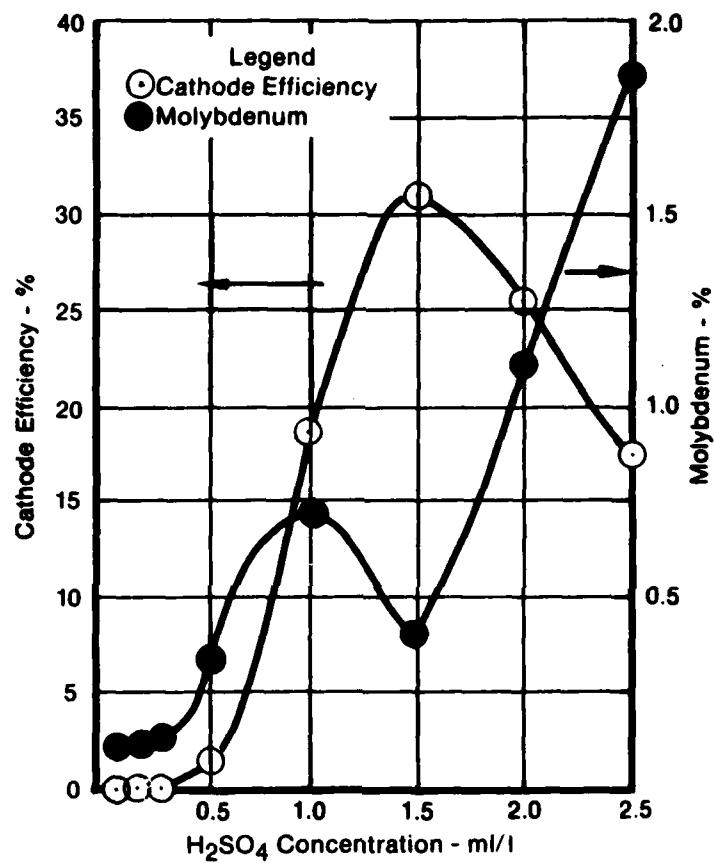
ml  $\text{H}_2\text{SO}_4/\text{l} = 2.0$   
% CE = 25.2  
% Mo = 1.1



ml  $\text{H}_2\text{SO}_4/\text{l} = 2.5$   
% CE = 17.6  
% Mo = 1.85

FD 144111

Figure 15. Specimens Pulse Plated in Chromium Trioxide-Based Solution, 0.1 to 2.5 ml/l Sulfuric Acid Concentration



FD 144112

Figure 16. Effect of Increasing Sulfuric Acid Concentration in Solution

Further additions of sulfuric acid produced unsatisfactory black coatings. At a concentration of 2.0 ml/l, the same needle-like appearance noted in several previous cases was observed. Figure 16 shows that above 1.0 ml/l all changes in cathode efficiency were accompanied by an inverse change in molybdenum content. The maximum cathode efficiency occurs at 1.5 ml/l. Unichrome, the self-regulating constituent used in all previous studies, contains a sulfate catalyst concentration corresponding to this level, indicating that Cr-Mo plating works best at the same sulfate concentration as the optimum for chromium deposition.

#### **Ammonium Molybdate ( $\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ Concentration**

This study utilized an aqueous solution containing 75 grams per liter (g/l) of ammonium molybdate and 300g/l of Unichrome. Ammonium molybdate was added to the solution in 20 gram (g) increments to determine its effect on the plating process. A pulse time of 40 ms "on", 40 ms "off" was used. Figure 17 shows the results obtained by SEM photos and Figure 18 presents a plot of cathode efficiency and molybdenum content as a function of ammonium molybdate concentration.

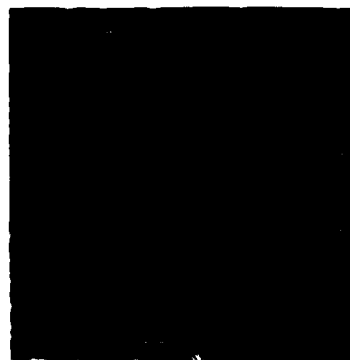
The SEM photos illustrate that the coatings produced at ammonium molybdate concentrations of 75 and 115 g/l are composed of randomly-shaped platelets. At 135 g/l, the surface consists of small platelets standing on edge. At 95 g/l, a rough surface with no distinct particle shape was found.

Figure 18 shows that when the ammonium molybdate concentration was increased, the molybdenum content of the deposit increased at the expense of cathode efficiency. This was expected, since the molybdate in solution occupies more and more space at the cathode as the concentration increases, thereby prohibiting some of the chromium in solution from being plated out. Very little of the molybdenum is actually electroplated. Most of that which is contained in the coating is transported by the migrating chromium ions. This explains why the cathode efficiency steadily decreases with increasing molybdate concentrations. These findings show that coatings with high molybdenum content can be obtained by increasing the ammonium molybdate concentration, but possible benefits must be weighed against the decrease in cathode efficiency.

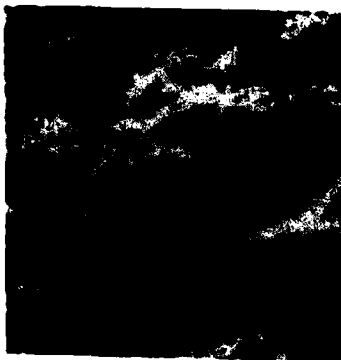
#### **Unichrome Concentration**

The initial composition of the solution in this study consisted of 150 g Unichrome, 75 g ammonium molybdate, and deionized water up to a volume of one liter. Unichrome was then added to the solution in 25 g increments to evaluate the effect on the coating produced. Pulse time was again selected at 40 ms "on" and 40 ms "off". Figure 19 shows SEM photos of the specimens and Figure 20 presents a plot of the variation in cathode efficiency and molybdenum content as the Unichrome concentration is increased.

(On- Off-Time = 40 ms; Mag: 1000X)



Ammonium Molybdate = 75 g/l  
% CE = 24.7  
% Mo = 0.72



Ammonium Molybdate = 95 g/l  
% CE = 19.7  
% Mo = 0.96



Ammonium Molybdate = 115 g/l  
% CE = 13.4  
% Mo = 1.64

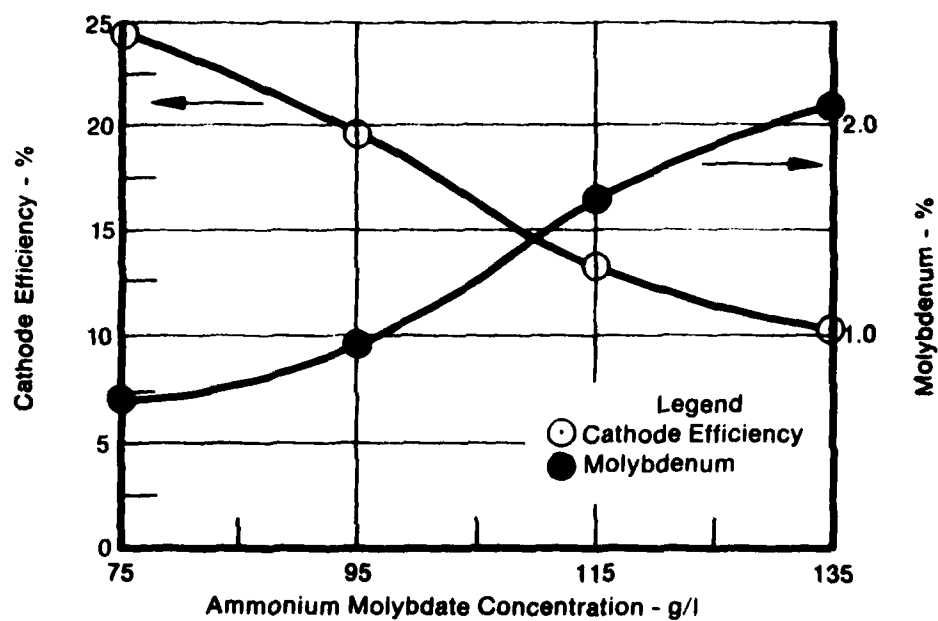


Ammonium Molybdate = 135 g/l  
% CE = 10.5  
% Mo = 2.06

FD 144122

*Figure 17. Specimens Pulse Plated in Unichrome-Based Solution, 75 to 135 g/l Ammonium Molybdate Concentration*





FD 144123

**Figure 18.** *Effect of Increasing Ammonium Molybdate Concentration in Solution*

(On - Off-Time = 40 ms; Mag: 1000X)



% CE = 1.6 % Mo = 6.05  
150 g Unichrome/l



% CE = 2.5 % Mo = 3.01  
175 g Unichrome/l



% CE = 7.1 % Mo = 1.51  
200 g Unichrome/l



% CE = 12.6 % Mo = 1.04  
225 g Unichrome/l



% CE = 17.1 % Mo = 1.16  
250 g Unichrome/l



% CE = 20.3 % Mo = 0.87  
275 g Unichrome/l



% CE = 21.9 % Mo = 0.82  
300 g Unichrome/l



% CE = 17.5 % Mo = 1.2  
325 g Unichrome/l



% CE = 18.0 % Mo = 1.05  
350 g Unichrome/l

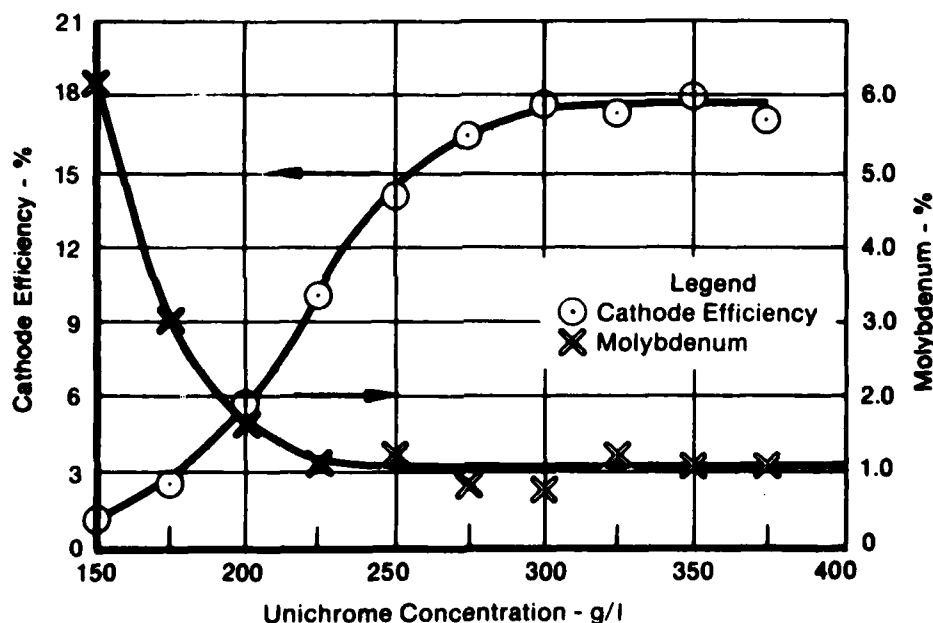


% CE = 17.3 % Mo = 1.04  
375 g Unichrome/l

FD 144124

Figure 19. Specimens Pulse Plated in Unichrome-Based Solution, 150 to 375 g/l Unichrome Concentration

The SEM photos show that nearly all of the coatings are composed of fine grains. Although several different shapes are seen (nodules, platelets, blocks), no drastic changes in surface topography occurred as the Unichrome concentration increased. Figure 20 shows high molybdenum contents were obtained at Unichrome concentrations of 150 g/l (6.05%) and 175 g/l (3.01%). This agrees with the findings above: that is, the molybdate concentration in solution is relatively high compared to the chromium ion concentration. Consequently, the cathode efficiencies at these concentrations are very low. At a concentration of 200 g of Unichrome per liter, the molybdenum content of the coating fell to 1.51%, but the cathode efficiency was only 5.8%, making this an undesirable concentration. At 225 g/l, the molybdenum content starts to level out, but major improvements in cathode efficiency occur until the Unichrome concentration reaches 275 g/l. At greater concentrations, the cathode efficiency also levels out. Beyond 300 g/l, no significant changes occur, suggesting that the saturation point of Unichrome had been reached.

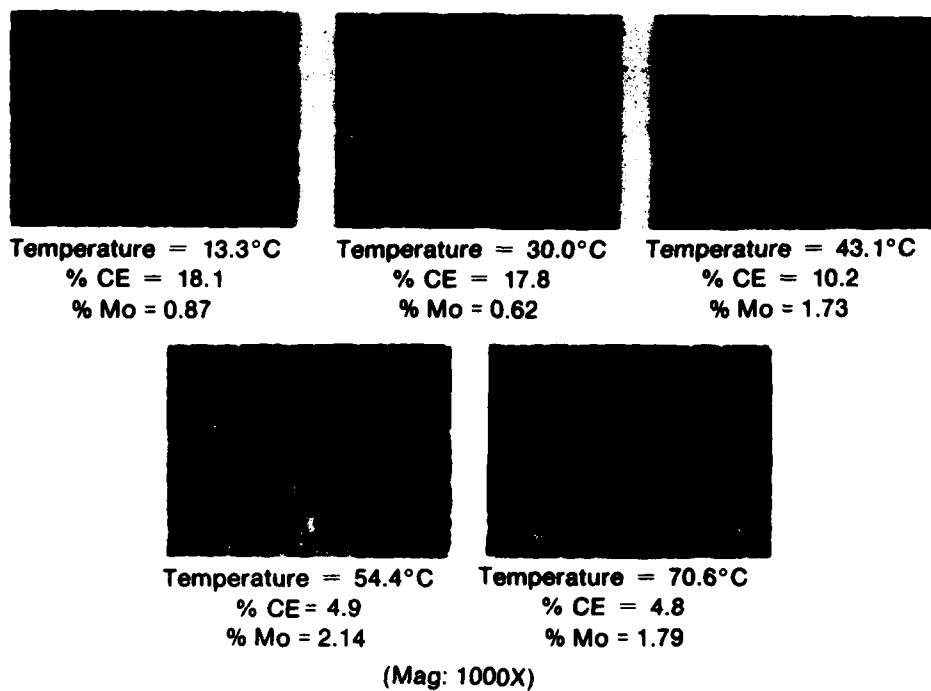


FD 144125

Figure 20. Effect of Increasing Chromic Acid Concentration in Solution

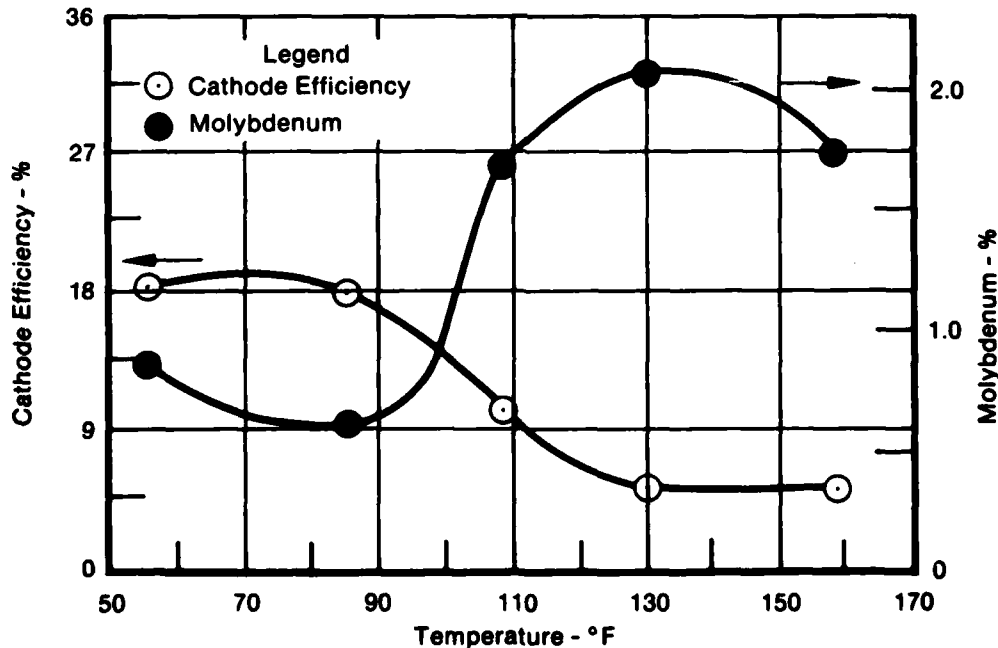
### Temperature

A test solution was prepared using 300 g/l Unichrome and 75 g/l ammonium molybdate. On/off-times were selected at 40 ms. Figure 21 shows the SEM photos obtained in this study and Figure 22 presents a plot of cathode efficiency and molybdenum content as a function of solution temperature.



FD 144126

Figure 21. Pulse Plated Specimens (On-Time = 40 ms/Off-Time = 40 ms) Showing Effect of Temperature on Deposition, 13.3 to 70.6°C



FD 144126

Figure 22. Effect of Solution Temperature on Cathode Efficiency and Molybdenum Content

The SEM photos reveal that a uniform coating was produced at all five temperatures. At 43.1°C (109.5°F), rectangular blocks are the predominant structures. At 70.6°C (159.0°F), rice-shaped grains interspersed with dark platelets were formed. The rest of the samples have a structure composed of nodular grains. The physical appearance of the samples indicated that the deposition process was proceeding unsatisfactorily at temperatures of 54.4°C (130.0°F) and 70.6°C (159.0°F) with cathode efficiencies also quite low. At 43.1°C (109.5°F), however, the appearance of the coating was good, the molybdenum content was relatively high, and the cathode efficiency was over 10%. Figure 22 shows a steady decrease in cathode efficiency and increase in molybdenum content between 30°C (86.0°F) and 54.5°C (130.0°F).

## **WEAR RESISTANCE**

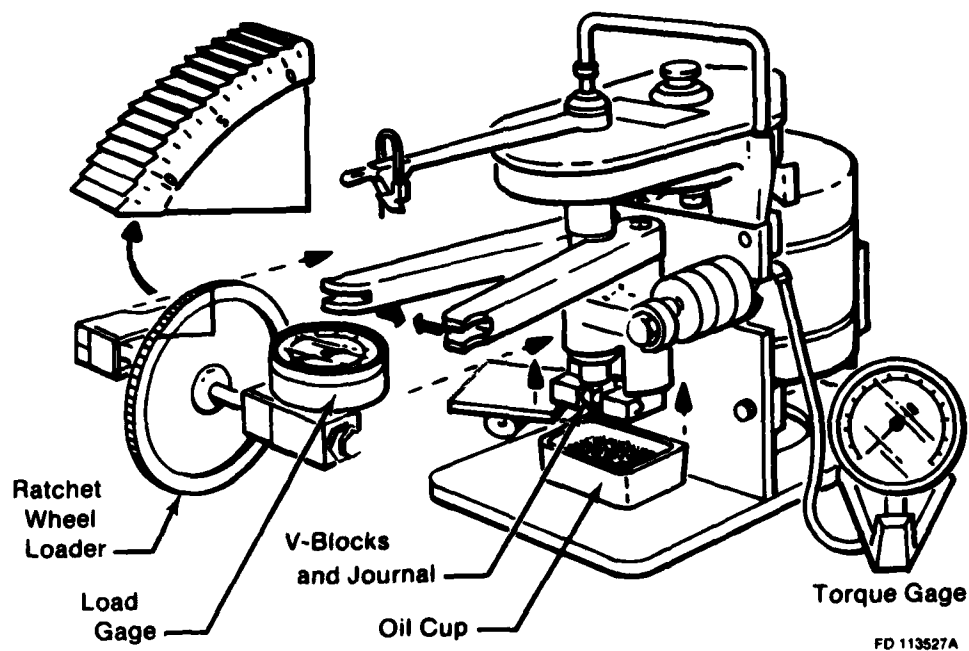
### **Wear Testing of Chromium-Molybdenum Plated Steel**

To assess the effect of pulse settings on the wear properties of the coating produced, eighteen different combinations of "on" and "off" times were used. These settings were chosen because SEM photographs had shown that they resulted in a wide variety of surface topographies on titanium. Test pins made of SAE3135 steel were plated at room temperature in a solution containing 300 g/l Unichrome and 75 g/l ammonium molybdate. A coating with a thickness of 0.00127 cm (0.0005 in.) was applied to each pin. These pins were tested with a Falex wear tester. This equipment is universally accepted as a method for screening wear couples. While results are not always correlatable to in-service conditions, it does provide a somewhat controlled method of materials comparison by providing fixed specimen geometry.

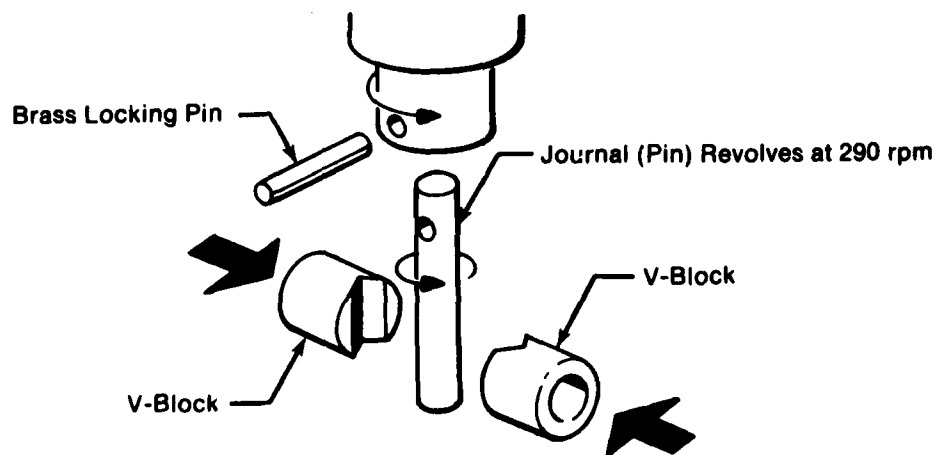
The Falex wear testing machine is shown in Figures 23 and 24. A 0.635-cm dia journal (pin) is rotated against two stationary V-blocks to give a four-line contact. The test pieces and their supporting jaws are immersed in the oil sample cup or run dry. Coatings or dry-film lubricants to be investigated are applied to the pins and/or V-blocks. The pin is rotated at 290 rpm and a load is applied to the V-blocks through a nutcracker action lever arm and spring gage. The load is applied by means of a ratchet wheel mechanism that also may be used to indicate the extent of wear like a micrometer. An 18-tooth advance on the ratchet wheel is equal to 0.00254 cm of wear. The entire load arm assembly is free to rotate about the main shaft, and friction developed during the test is shown in inch-pounds on the torque gage.

In the test method selected, the V-block load was increased incrementally according to the schedule given in ASTM-D-2625 Procedure A. It consists of running two stationary V-block specimens against a rotating pin until a sharp increase of 1.13 N-m (10 in.-lb) in steady-state torque or pin breakage is experienced. A prescribed schedule of jaw load application and dwell time on the rotating pin is followed: 3 min at 1330 N plus 1 min at 2220 N plus 1 min at 3330 N plus 4450 N until failure.

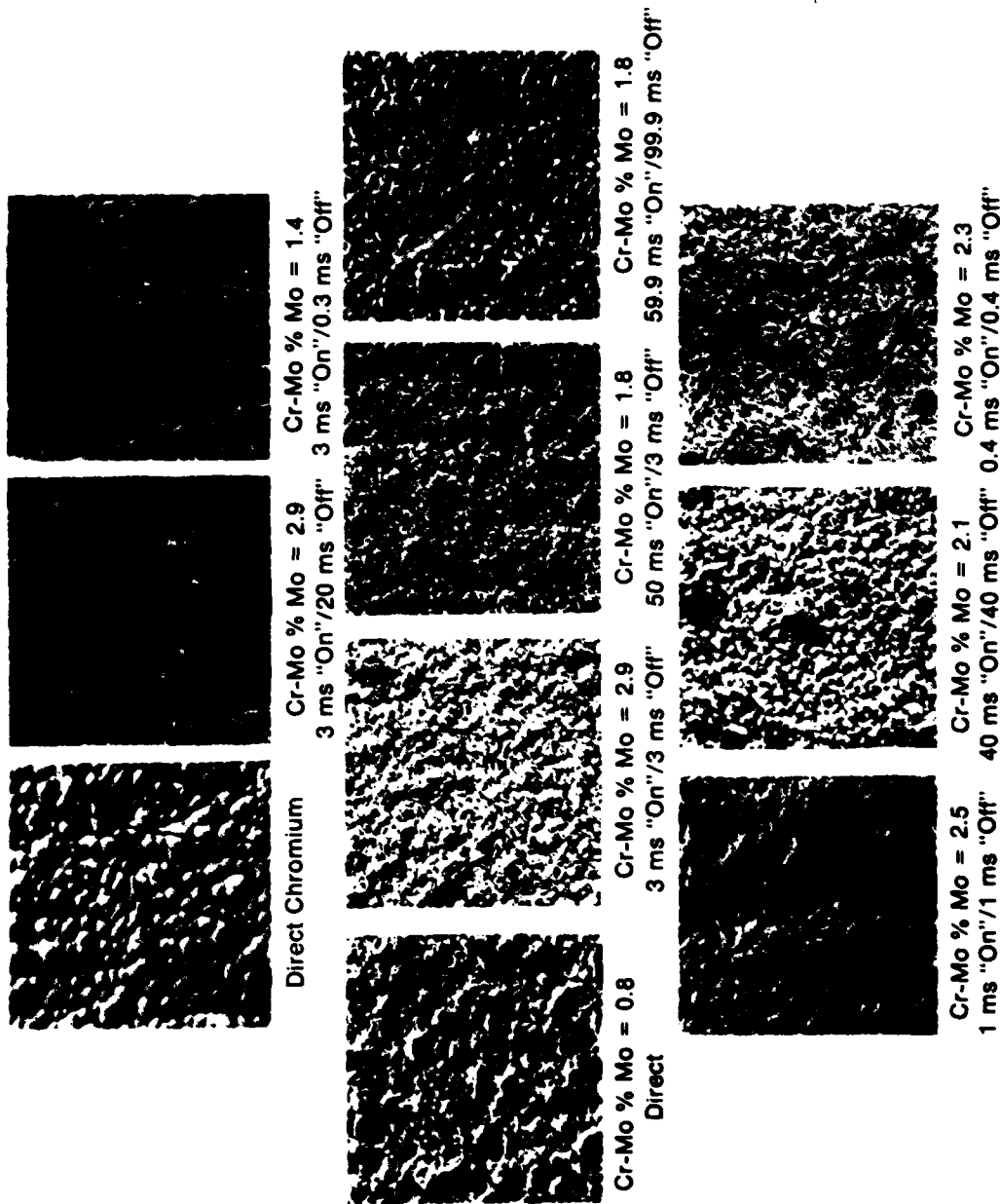
For this study, the oil cup was filled with MIL-L-23699 aircraft turbine engine lubricant. To enable a large number of settings to be evaluated, the V-blocks (made of AISI 1137 steel) were left uncoated. Table 2 details the results. Since all pins satisfactorily completed the testing at 1330, 2220, and 3330 N, only the run time at 4450 N is shown. The pins plated at 3 ms "on", 20 ms "off" and 3 ms "on", 3 ms "off" did not fail after 30 min at 1000 lb. Figure 25 shows SEM photos of the eight longest-wearing pulse-plated specimens, and of direct-plated chromium-molybdenum and chromium.



**Figure 23. Schematic Diagram of Falex Lubricant Tester**



**Figure 24. Exploded View of V-Blocks and Journal Arrangement, Falex Lubricant Tester**



FD 163599

Figure 25. SEM Photographs of Wear Specimen Coatings

TABLE 2. FALEX WEAR TEST USING MIL-L-23699 OIL (COATED STEEL PINS/UNCOATED STEEL V-BLOCKS) (Phase I)

| <i>Pulse Setting<br/>(ms "On"/ms "Off")</i> | <i>Minutes to Failure<br/>at 4450N<br/>(1000 lbf)</i> | <i>Mo<br/>(%)</i> |
|---|---|-------------------|
| 3/20  | DNF <sup>1</sup>                                      | 2.9               |
| 3/0.3                                       | DNF   | 1.4               |
| Direct                                      | 20.1  | 0.8               |
| 3/3   | 14.3  | 2.9               |
| 50/3  | 13.6  | 1.8               |
| 59.9/99.9                                   | 13.4  | 1.8               |
| 1/1   | 13.4  | 2.5               |
| 40/40                                       | 11.5  | 2.1               |
| 0.4/0.4                                     | 11.1  | 2.3               |
| 10/80                                       | 9.4   | 1.9               |
| 60/80                                       | 8.6   | 1.1               |
| 99.9/39.9                                   | 8.1   | 1.85              |
| 7/3   | 6.5   | 2.9               |
| 49.9/99.9                                   | 6.4   | 1.65              |
| 20/3  | 5.3   | 2.0               |
| 60/20                                       | 4.6   | 2.5               |
| 99.9/99.9                                   | 1.4   | 1.8               |
| 0.6/3                                       | Failed <sup>2</sup>                                   | 2.9               |
| 0.9/3                                       | Failed  | 3.0               |

Notes:

<sup>1</sup> "DNF" indicates samples did not fail after 30 min at this load.

<sup>2</sup> "Failed" indicates samples failed while the jaw load was being increased to this level.

Surprisingly, pins direct-plated with chromium did not fail within 30 min, but the chromium plate was badly cracked, as seen in Figure 25. Possibly, oil filled the cracks reducing friction and enabling the pins to last significantly longer than was anticipated. However, these cracks increased base metal corrosion by providing a pathway for aggressive ions, such as chloride, to the underlying substrate. To find the coating with the best wear resistance, wear testing was repeated without oil. The results are shown in Table 3. All three specimens which exhibit cracked surfaces in Figure 25 (direct chromium, Cr-Mo at 3 ms "on"/20 ms "off", and Cr-Mo at 40 ms "on"/40 ms "off") did not do well in this test, lending support to the theory that cracks prolonged wear life when testing in oil.



**TABLE 3. FALEX WEAR TEST USING NO LUBRICATION  
(COATED STEEL PINS/UNCOATED STEEL  
V-BLOCKS) (Phase I)**

| Pulse Setting<br>(ms "On"/ms "Off") | Minutes to Failure at Specified Loads |                    |                    |                     |
|-------------------------------------|---------------------------------------|--------------------|--------------------|---------------------|
|                                     | 1330N<br>(300 lbf)                    | 2220N<br>(500 lbf) | 3330N<br>(750 lbf) | 4450N<br>(1000 lbf) |
| 59.9/99.9                           | 3                                     | 1                  | 1                  | 2.6                 |
| 3/3                                 | 3                                     | 1                  | 1                  | Failed              |
| 3/0.3                               | 3                                     | 1                  | 0.9                |                     |
| 50/3                                | 3                                     | 1                  | 0.7                |                     |
| 3/20                                | Failed                                |                    |                    |                     |
| Direct                              | Failed                                |                    |                    |                     |
| 1/1                                 | Failed                                |                    |                    |                     |
| 40/40                               | Failed                                |                    |                    |                     |
| 0.4/0.4                             | Failed                                |                    |                    |                     |
| Chromium (Direct)                   | Failed                                |                    |                    |                     |

To evaluate the effect of plating solution temperature on the wear properties of the coatings, two studies were conducted. In the first study, pins were direct-plated with chromium-molybdenum at various solution temperatures, with the test results summarized in Table 4. All temperature rises were accompanied by an increase in wear life. However, cathode efficiency has been shown to drop below 10% at temperatures greater than 43°C (110°F), showing that improved wear resistance must be weighed against reduced plating efficiency. To determine if wear life would be improved at various pulse settings as temperature is increased, pins were plated at 43°C using the four best settings from Table 3 plus direct chromium-molybdenum. Table 5 shows the test results and Figure 26 displays the SEM photographs of the coatings. To facilitate comparison, wear results from pins plated at room temperature are included in Table 5. It is evident that the increase in temperature caused the molybdenum content of the coatings to rise by an average of 66%. Wear life was longer at the higher plating temperature for four of the five settings. It is quite likely that the increased molybdenum content was responsible for the prolonged wear life. In addition, comparison of the SEM photographs in Figures 25 and 26 reveal that, for a given pulse setting, the grain size of the coatings increased as the temperature of the plating solution was increased from 25° to 43°C. This increased grain size may also have been a factor in prolonging the wear life.

**TABLE 4. FALEX WEAR TEST  
USING MIL-L-23699  
OIL (COATED STEEL  
PINS/UNCOATED  
STEEL V-BLOCKS)  
(Phase II)**

| Solution<br>Temperature<br>(°C) | Minutes to Failure<br>at 4450N<br>(1000 lbf) |
|---------------------------------|--|
| 25                              | 20.1   |
| 43                              | 42.5   |
| 49                              | 78.2   |
| 54                              | DNF after 100 min                            |



59.9 ms "On"/99.9 ms "Off"



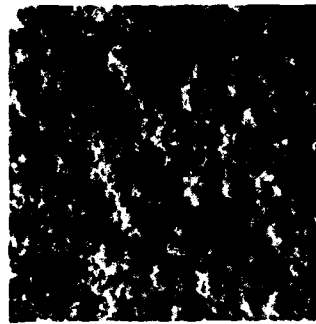
3 ms "On"/3 ms "Off"



3 ms "On"/0.3 ms "Off"



50 ms "On"/3 ms "Off"



Direct Current

FD 169911  
791807

Figure 26. SEM Photographs of Wear Specimen Coatings Plated at 43°C

**TABLE 5. FALEX WEAR TEST USING NO LUBRICATION  
(COATED STEEL PINS/UNCOATED STEEL V-BLOCKS)  
(Phase II)**

| Pulse Setting<br>(ms "On" ms "Off") | Plating<br>Temperature | Mo<br>(%) | Minutes to Failure at Specified Loads |                    |                    |                     |
|-------------------------------------|------------------------|-----------|---------------------------------------|--------------------|--------------------|---------------------|
|                                     |                        |           | 1330N<br>(300 lbf)                    | 2220N<br>(500 lbf) | 3330N<br>(750 lbf) | 4450N<br>(1000 lbf) |
| 59.9-99.9                           | 25°C                   | 1.8       | 3                                     | 1                  | 1                  | 2.6                 |
|                                     | 43°C                   | 2.4       | 3                                     | 1                  | 1                  | 3.6                 |
| 3-3                                 | 25°C                   | 2.9       | 3                                     | 1                  | 1                  | Failed              |
|                                     | 43°C                   | 4.0       | Failed                                |                    |                    |                     |
| 3-0.3                               | 25°C                   | 1.4       | 3                                     | 1                  | 0.9                |                     |
|                                     | 43°C                   | 2.9       | 3                                     | 1                  | 1                  | Failed              |
| 50-3                                | 25°C                   | 1.8       | 3                                     | 1                  | 0.7                |                     |
|                                     | 43°C                   | 2.2       | 3                                     | 1                  | 1                  | 2.6                 |
| Direct                              | 25°C                   | 0.8       | Failed                                |                    |                    |                     |
|                                     | 43°C                   | 2.9       | 3                                     | 1                  | 1                  | 1.5                 |

#### **Wear Testing of Pulse-Plated Chromium on Steel**

Steel pins were plated with chromium using direct current and pulse settings of 59.9 ms "on", 99.9 ms "off"; 3 ms "on", 3 ms "off"; 3 ms "on", 0.3 ms "off"; and 50 ms "on", 3 ms "off". The plating solution contained 300 g/l Unichrome and was maintained at a temperature of 49°C (120°F). None of the pulse settings produced a coating which wore better than direct-plated chromium.

#### **Wear Testing of Chromium-Molybdenum Plated Titanium**

Test pins made of Ti 6Al-4V were plated at 43°C (110°F) in a solution containing 300 g/l Unichrome and 75 g/l ammonium molybdate. In addition to the direct current, the four pulse settings which produced the coatings with the best wear resistance on steel were used. A coating with a thickness of 0.00127 cm (0.0005 in.) was applied to each pin. Five diffusion heat treat cycles were evaluated for each pulse setting. Table 6 shows these cycles along with the corresponding wear data. The pins were tested with a Falex wear tester using uncoated AISI1137 steel V-blocks and no lubrication. A pulse setting of 3 ms "on", 3 ms "off" emerged as the most effective setting in producing a wear resistant surface. A heat treat cycle of 3 hr at 760°C (1400°F) followed by 6 hr at 538°C (1000°F) optimized the wear life of this coating.

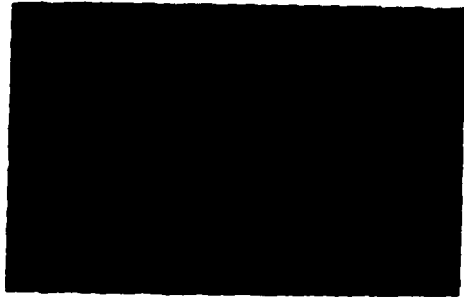
**TABLE 6. FALEX WEAR TEST USING NO LUBRICATION (COATED  
TITANIUM PINS/UNCOATED STEEL V-BLOCKS)**

| Pulse Setting<br>(ms "On" ms "Off") | Heat-Treat<br>Cycle              | Minutes to Failure at Specified Loads |                    |                    |                     |
|-------------------------------------|----------------------------------|---------------------------------------|--------------------|--------------------|---------------------|
|                                     |                                  | 1330N<br>(300 lbf)                    | 2220N<br>(500 lbf) | 3330N<br>(750 lbf) | 4450N<br>(1000 lbf) |
| 3-3                                 | 3 hr at 760°C                    | 3                                     | 1                  | 0.5                |                     |
|                                     | 3 hr at 760°C<br>+ 6 hr at 538°C | 3                                     | 1                  | 1                  | 1.6                 |
|                                     | 1 hr at 871°C                    | Failed                                |                    |                    |                     |
|                                     | 1 hr at 871°C<br>+ 6 hr at 593°C | 0.5                                   |                    |                    |                     |
|                                     | 1 hr at 982°C<br>+ 8 hr at 593°C | Failed                                |                    |                    |                     |

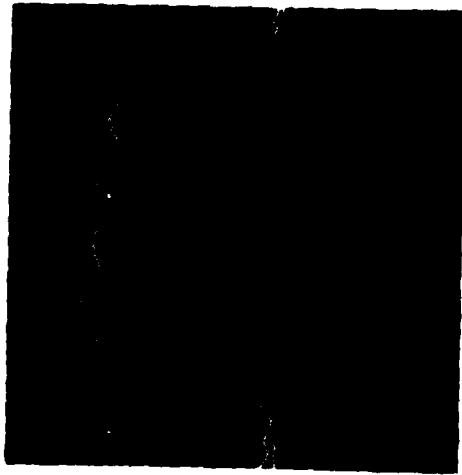
**TABLE 6. FALEX WEAR TEST USING NO LUBRICATION (COATED TITANIUM PINS/UNCOATED STEEL V-BLOCKS) (Continued)**

| <i>Pulse Setting<br/>(ms "On" ms "Off")</i> | <i>Heat-Treat<br/>Cycle</i>      | <i>Minutes to Failure at Specified Loads</i> |                            |                            |                             |
|---|----------------------------------|--|----------------------------|----------------------------|-----------------------------|
|   |                                  | <i>1330N<br/>(300 lbf)</i>                   | <i>2220N<br/>(500 lbf)</i> | <i>3330N<br/>(750 lbf)</i> | <i>4450N<br/>(1000 lbf)</i> |
| 59.9 99.9                                   | 3 hr at 760°C                    | 1.5  |                            |                            |                             |
|   | 3 hr at 760°C<br>+ 6 hr at 538°C | 1  |                            |                            |                             |
|   | 1 hr at 871°C                    | Failed                                       |                            |                            |                             |
|   | 1 hr at 871°C<br>+ 6 hr at 593°C | 0.4  |                            |                            |                             |
|   | 1 hr at 982°C<br>+ 8 hr at 593°C | Failed                                       |                            |                            |                             |
| 50 3  | 3 hr at 760°C                    | Failed                                       |                            |                            |                             |
|   | 3 hr at 760°C<br>+ 6 hr at 538°C | 0.1  |                            |                            |                             |
|   | 1 hr at 871°C                    | Failed                                       |                            |                            |                             |
|   | 1 hr at 871°C<br>+ 6 hr at 593°C | Failed                                       |                            |                            |                             |
|   | 1 hr at 982°C<br>+ 8 hr at 593°C | Failed                                       |                            |                            |                             |
| 3 0.3                                       | 3 hr at 760°C                    | 3  | 0.3                        |                            |                             |
|   | 3 hr at 760°C<br>+ 6 hr at 538°C | 0.7  |                            |                            |                             |
|   | 1 hr at 871°C                    | Failed                                       |                            |                            |                             |
|   | 1 hr at 871°C<br>+ 6 hr at 593°C | Failed                                       |                            |                            |                             |
|   | 1 hr at 982°C<br>+ 8 hr at 593°C | Failed                                       |                            |                            |                             |
| Direct                                      | 3 hr at 760°C                    | Failed                                       |                            |                            |                             |
|   | 3 hr at 760°C<br>+ 6 hr at 583°C | Failed                                       |                            |                            |                             |
|   | 1 hr at 871°C                    | Failed                                       |                            |                            |                             |
|   | 1 hr at 871°C<br>+ 6 hr at 593°C | Failed                                       |                            |                            |                             |
|   | 1 hr at 982°C<br>+ 8 hr at 593°C | Failed                                       |                            |                            |                             |

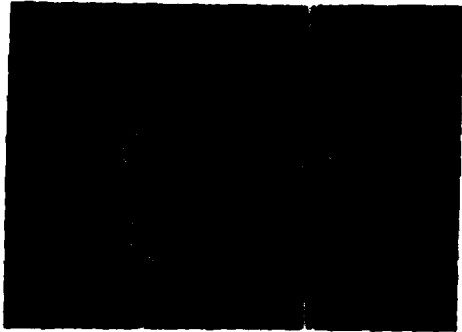
Figure 27 shows SEM photographs of the coatings produced at various pulse cycles after diffusion heat treatment for 3 hr at 760°C (1400°F) followed by 6 hr at 538°C (1000°F). Superimposed over each photograph is a plot of the chromium content across the coating and diffusion layer. These photographs, together with the wear data from Table 6 show that there is an inverse



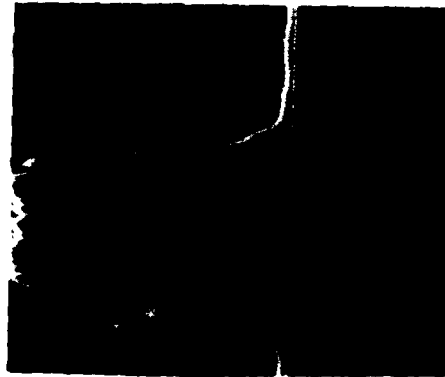
3 ms "On"/3 ms "Off"



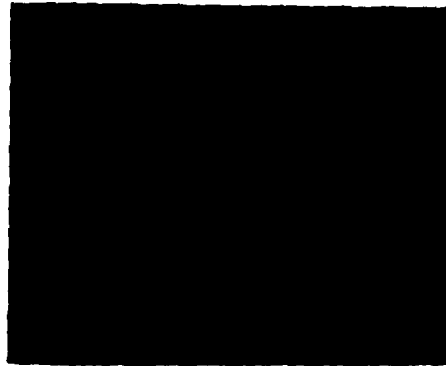
59.9 ms "On"/99.9 ms "Off"



3 ms "On"/0.3 ms "Off"



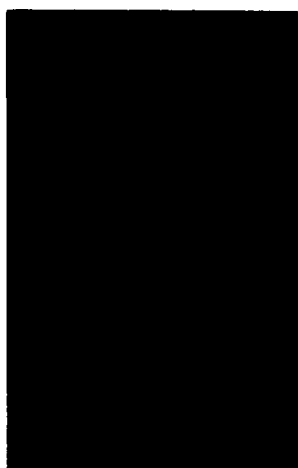
50 ms "On"/3 ms "Off"



Direct Current

Heat Treat Cycle: 3 hr at 760°C + 6 hr at 538°C  
Mag: 1000X

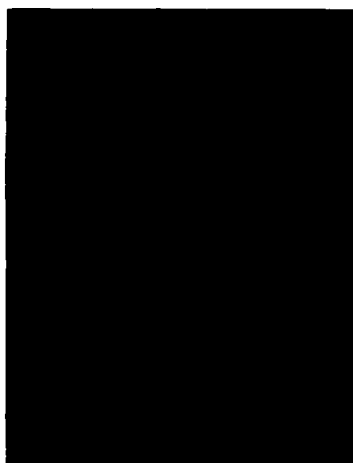
*Figure 27. Diffusion of Chromium into Ti 6Al-4V at Various Pulse Settings*



3 hr at 760°C  
+ 6 hr at 538°C



1 hr at 871°C  
+ 6 hr at 593°C



3 hr at 760°C



1 hr at 871°C

Pulse Setting: 3 ms "On"/3 ms "Off"  
Mag: 1000X

FD 173103

*Figure 28. Diffusion of Chromium into Ti 6Al-4V at Various Heat Treat Cycles*

relationship between the depth of the diffusion layer and the wear life in this case. In Figure 28, coatings produced with a pulse setting of 3 ms "on", 3 ms "off" are shown after various heat treat cycles. Once again, the inverse relationship between diffusion depth and wear life can be seen. As expected, increased heat-treat temperature led to greater diffusion depth. None of the pins heat treated at temperatures greater than 760°C lasted more than 30 sec at a load of only 1330N. It is possible that brittle Cr<sub>2</sub>Ti intermetallics were formed at these high temperatures leading to early pin failure due to degradation of mechanical strength properties.

To assess the effect of various heat-treat cycles on the wear properties of chromium-molybdenum plated Ti 8Al-1Mo-1V, pins were fabricated from bar stock and plated at a pulse setting of 3 ms "on", 3 ms "off". The pins were tested without lubrication. Five heat-treat cycles were evaluated, as shown in the SEM photographs in Figure 29. As before, the diffusion depth increased as the heat-treat temperature was raised. However, the best wear resistance was obtained after heat treatment at 982°C (1800°F) for one hr followed by eight hr at 593°C (1100°F). This cycle produced the deepest diffusion layer and implies that a greater depth of diffusion is required to gain suitable adhesion of chromium-molybdenum to Ti 8-1-1 than that required for Ti 6-4. It should be noted, however, that the best-wearing Ti 8-1-1 pins lasted only 0.66 min at a load of 1330N and all others failed before reaching this load. This lack of numerical data makes it impossible to pinpoint the relationship between diffusion depth and wear resistance for Ti 8-1-1.

#### Wear Testing With Dry-Film Lubricant

Steel pins plated with chromium-molybdenum at 59.9 ms "on", 99.9 ms "off" and with direct chromium, and Ti 6-4 pins plated with Cr-Mo at 3 ms "on", 3 ms "off" and with direct chromium were coated with a molybdenum disulfide dry-film lubricant.<sup>1</sup> This substantially improved the wear life of all the coatings. After one hr at a load of 4450 N in the Falex wear tester, none of the pins had failed, and micrometer measurements showed that a substantial amount of coating remained on each pin. The chromium-molybdenum coatings showed considerably less wear than those of chromium on both steel and titanium. Table 7 details the results of this investigation.

TABLE 7. FALEX WEAR TEST USING A DRY-FILM LUBRICANT

| Pin/Coating    | Pulse Setting<br>(ms "On" ms "Off") | Pin Diameter (cm) |                          |                        | % of Total<br>Coating Removed |
|----------------|-------------------------------------|-------------------|--------------------------|------------------------|-------------------------------|
|                |                                     | Initial           | After Plating<br>and DFL | After 1 hr<br>at 4450N |                               |
| Steel/Cr-Mo    | 59.9/99.9                           | 0.6350            | 0.6391                   | 0.6379                 | 29.3                          |
| Steel/Cr       | Direct                              | 0.6350            | 0.6388                   | 0.6369                 | 50.0                          |
| Titanium/Cr-Mo | 3/3                                 | 0.6172            | 0.6198                   | 0.6193                 | 19.2                          |
| Titanium/Cr    | Direct                              | 0.6172            | 0.6198                   | 0.6181                 | 65.4                          |

#### CORROSION RESISTANCE

Roller bearings of AMS6490 steel were plated with 0.00254 cm (0.001 in.) of direct chromium, direct chromium-molybdenum, and pulsed chromium-molybdenum at pulse times of 3 ms "on", 3 ms "off"; 3 ms "on", 0.3 ms "off"; 50 ms "on", 3 ms "off"; and 59.9 ms "on", 99.9 ms "off". These bearings were then subjected to salt-spray corrosion testing for eight hr in accordance with the procedure outlined in ASTM B-117. Figure 30 shows the bearings after completion of the testing. The ends of each bearing were masked during the testing and, therefore, indicate the original appearance of the coatings. The chromium plating appears more corrosion resistant than some of the chromium-molybdenum, most notably that pulse plated at 3 ms "on", 3 ms "off".



1 hr at 760°C  
+ 6 hr at 538°C



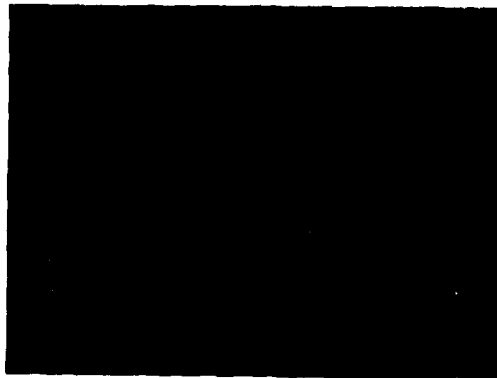
1 hr at 871°C  
+ 6 hr at 593°C



1 hr at 982°C  
+ 8 hr at 593°C



1 hr at 871°C



1 hr at 982°C

Pulse Setting: 3 ms "On"/3 ms "Off"  
Mag: 1000X

Figure 29. Diffusion of Chromium into Ti 8Al-1Mo-1V at Various Heat Treat Cycles





FD 177408

*Figure 30. Coated AMS6490 Bearings After 8-Hour Salt Spray Test*

Referring back to Figure 27, it can be seen that this coating is more porous than those produced at the other settings. The coating exhibiting the least corrosion is that obtained at a pulse setting of 50 ms "on", 3 ms "off", suggesting that the best way to apply the chromium-molybdenum coating to steel is to plate half of the desired thickness at 50 ms "on", 3 ms "off" and then change the setting to 59.9 ms "on", 99.9 ms "off" (for maximum wear resistance) for the remainder of coating thickness desired.

### LOW-CYCLE FATIGUE

To evaluate the effects of the chromium-molybdenum plating/diffusion procedure on the low-cycle fatigue (LCF) strength at Ti 8Al-1Mo-1V, specimens were machined from AMS4972 bar stock, as shown in Figure 31. These specimens were plated and heat treated in accordance with the procedures outlined in Appendix A. LCF testing was conducted at temperatures of 260°C (500°F) and 500°C (932°F). Each test cycle consisted of increasing tensile strain on a specimen from 0 to 1.0% and then returning the strain to 0, resulting in an average strain of 0.5%. These cycles were run at a rate of 10 cycles per minute until the specimen failed. Figure 32 shows the test results for coated and uncoated specimens at both temperatures.

The dashed lines in Figure 32 show the changes in mean cycles to failure as the temperature was raised from 260° to 500°C, while the solid lines show the changes in the 97.5% lower bound. This value represents a point above which the cycles to failure of 97.5% of the specimens in a lot can be expected to fall. At 260°C (500°F), the coated specimens exhibited a 56.7% decrease in mean LCF and a 72.4% decrease in the lower bound when compared to the uncoated specimens. At 500°C (932°F), coated specimens had a 22.2% lower mean fatigue life than uncoated samples, but the lower bound was 34.0% higher than that for the uncoated specimens. Increasing the temperature from 260° to 500°C resulted in a 64.9% decrease in mean LCF for uncoated specimens as opposed to a 36.8% decrease for coated specimens. This temperature change also decreased the lower bound for uncoated specimens by 70.6%, but increased that for coated specimens by 42.2%.

These results suggest that the chromium-molybdenum plating process is highly detrimental to the LCF strength of Ti 8-1-1, but the oxidation resistance afforded by the coating slows the rate of fatigue strength degradation as the temperature is increased. At temperatures above 500°C, it is likely that the corresponding rates of fatigue strength degradation are such that the LCF life of coated specimens is equivalent to that of uncoated specimens.

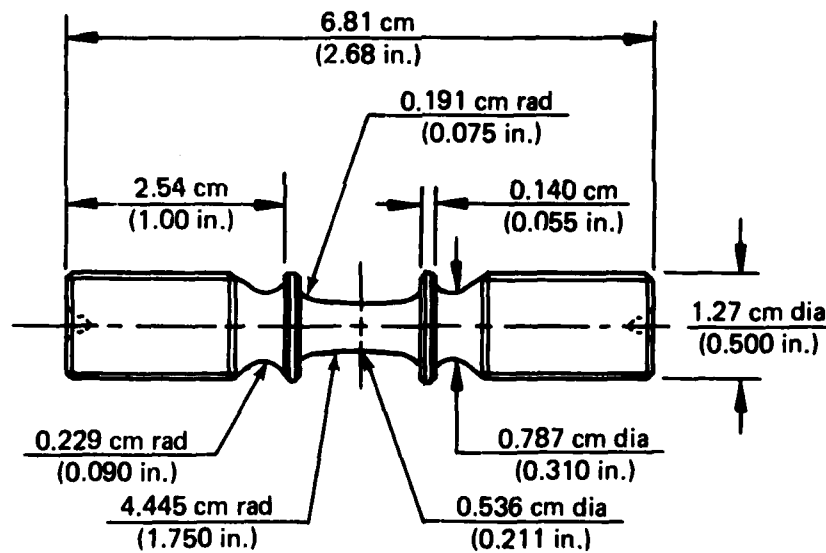
### COMPONENT TESTING

A Ti 8Al-1Mo-1V (AMS 4972) third-stage fan blade from a P&WA TF30 gas turbine engine was selected for component testing with chromium-molybdenum coatings. This part experienced galling problems during engine operations. As a potential solution to this problem, chromium-molybdenum (Cr-Mo) was electroplated onto the root of one of the blades using a conforming anode and the plating/diffusion process outlined in Appendix A. Figure 33 shows the plated, heat treated blade. After an application of Dow Corning 3400 A<sup>1</sup> dry-film lubricant to the Cr-Mo coating, P&WA compared the galling resistance of this coating system with that of an uncoated blade and a bill of material (BOM) configuration that uses the dry-film lubricant alone. Testing was conducted with the blade roots restrained in a TF30 third-stage fan blade broached fixture and jack bolt, loaded from the rear. Stressing one blade to failure at room temperature established the maximum first bending mode stress locations. This determined the stress level at which the galling tests were to be conducted. Each blade was vibrated at  $1.7 \times 10^4 \text{ N/cm}^2$  to a total of  $10^7$  cycles at room temperature in the first bending mode of vibration, with a jack bolt load that would result in galling of the root. Testing the blades in individual broach blocks ensured that each blade would be subjected to the same test conditions.

<sup>1</sup>Dow Corning Corporation, 3400A dry-film lubricant

After testing, each blade underwent a quantitative galling evaluation. Based on a comparison of the percent of contact area which was galled, the chromium-molybdenum/dry-film lubricant system proved far superior to the dry-film lubricant alone. However, the results of the uncoated blade compared closely with those of the Cr-Mo/dry-film lubricant system. The BOM blade root exhibited galling over approximately 20% of the contact surface area on the convex side, compared to 10% for the other two configurations. On the concave side, the BOM blade root galled over 30% of the contact area as opposed to 8% for the uncoated root and only 5% for the Cr-Mo/dry-film lubricant-coated blade root.

Although the use of a Cr-Mo/dry-film lubricant coating appeared to significantly enhance the galling resistance of the blade root compared to the BOM blade, this result is challenged by the fact that the uncoated blade fared nearly as well in the test. It is inconceivable that the use of a dry-film lubricant alone could actually decrease the galling resistance of the blade material. Moreover, this test represented only a simulation of in-flight conditions and results were based on only one blade for each of the three configurations. Engine testing of titanium blades in the next phase of the program will provide more comprehensive data on galling resistance.



FD 168228

Figure 31. Low-Cycle Fatigue Specimen

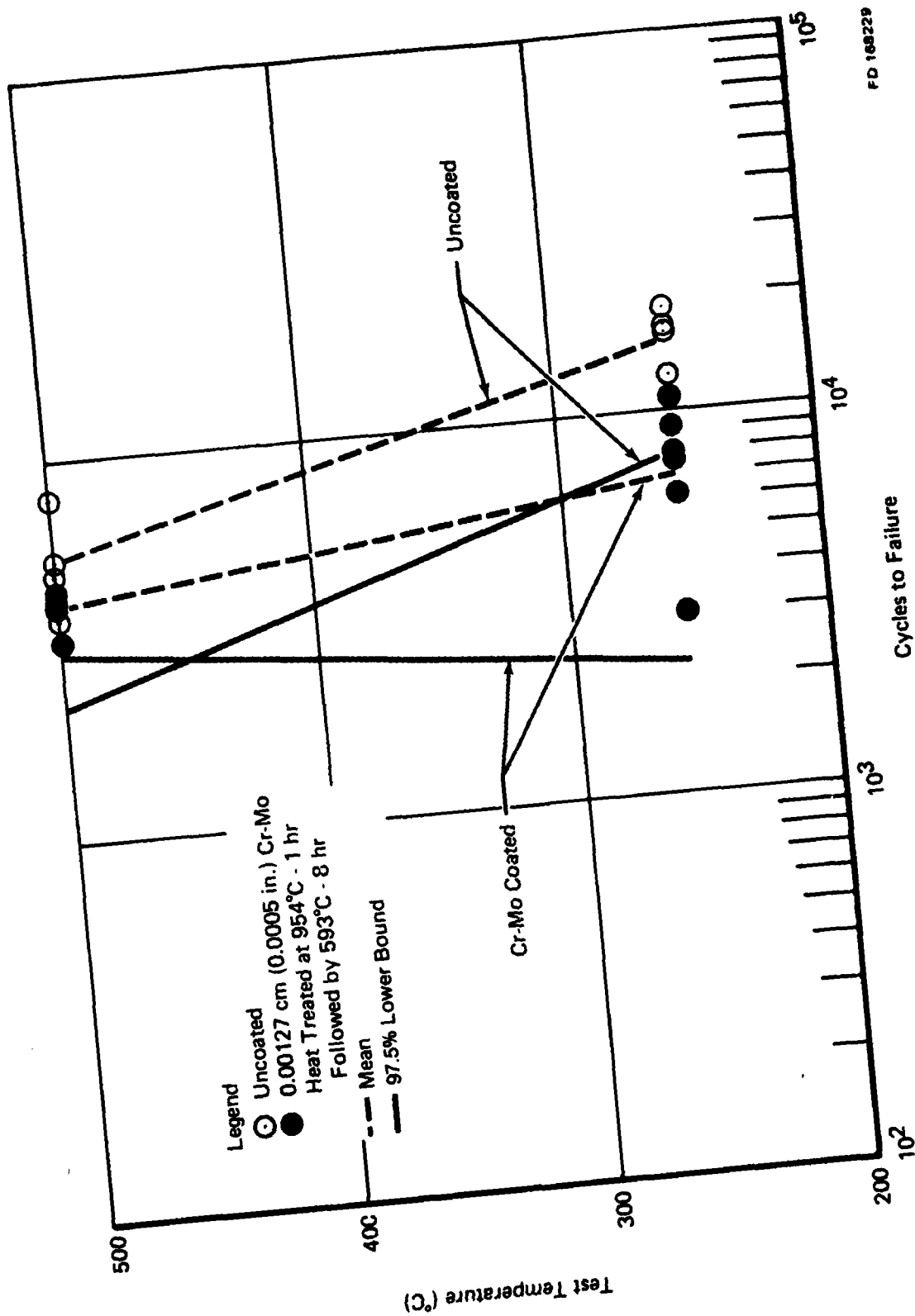
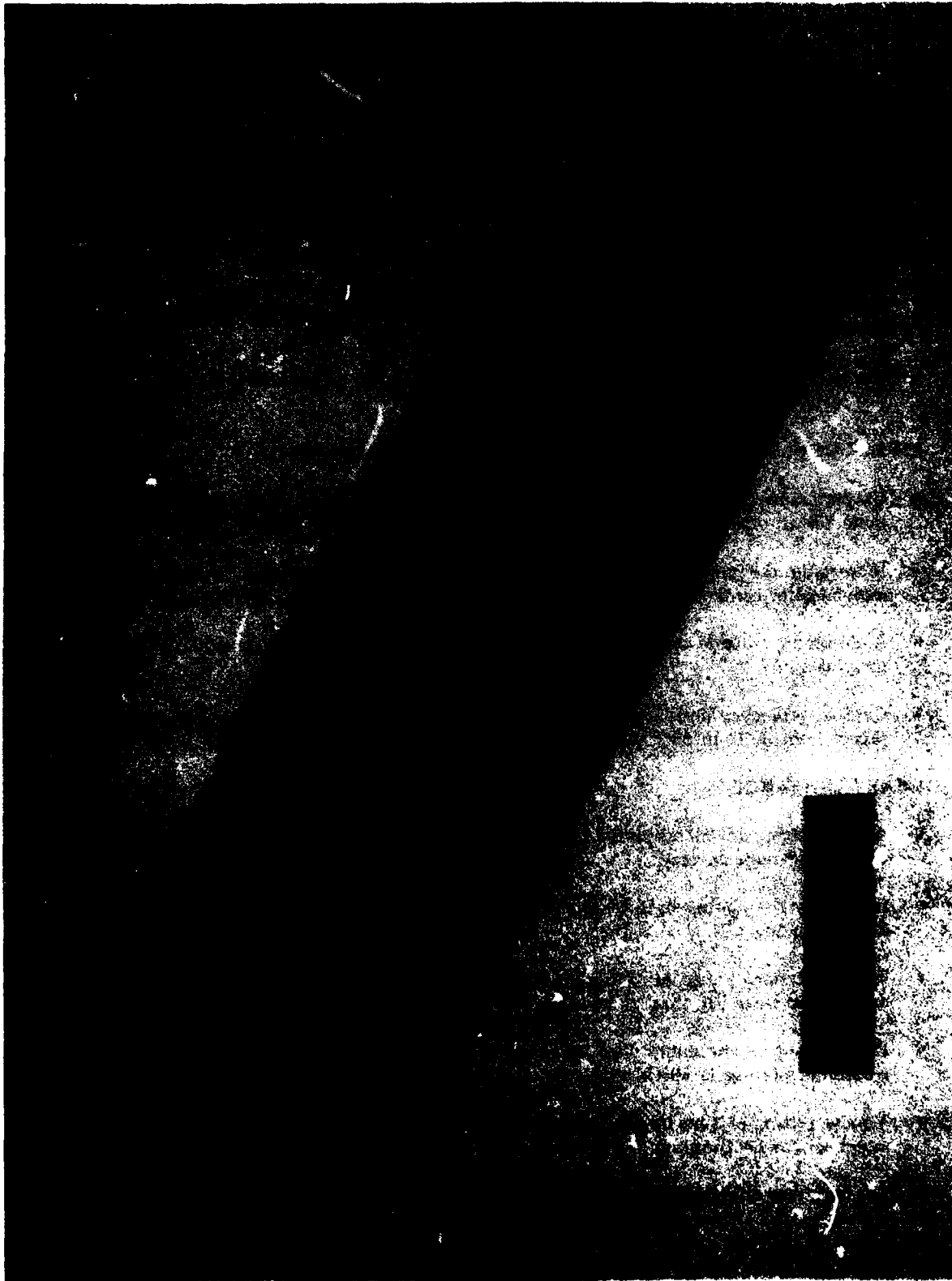


Figure 32. Effect of Electrodeposited Cr-Mo on Low Cycle Fatigue



FD 188227

*Figure 33. TF-30 Third Stage Fan Blade with Cr-Mo Coated Root*

### **SECTION III CONCLUSIONS**

#### **EFFECT OF PULSE SETTINGS**

- At a given on-time, a change in off-time will result in a change in cathode efficiency and an inverse change in the molybdenum content.
- Variations in off-time are more critical than variations in on-time.
- High cathode efficiency and low molybdenum content result at high duty cycles (on-time / (on-time + off-time) > 0.7).
- For a given on-time, hardness decreases with increasing off-time to a minimum value of 210 VHN.

#### **EFFECT OF PLATING BATH COMPOSITION AND TEMPERATURE**

- Chromium-molybdenum plating works best at the same sulfate concentration as the optimum for chromium deposition (100g chromium trioxide to 1g sulfuric acid).
- Increasing the ammonium molybdate concentration of the plating bath increases the molybdenum content of the deposits, but at the expense of cathode efficiency.
- A chromium trioxide concentration of 300g is optimum for efficient deposition of chromium-molybdenum.
- Cathode efficiency decreases and molybdenum content increases as plating temperature is raised from 30°C to 55°C with the optimum reached at 43°C.

#### **WEAR RESISTANCE**

- A pulse setting of 59.9 ms on-time, 99.9 ms off-time maximizes the wear resistance of chromium-molybdenum on steel.
- Increasing the plating bath temperature improves the wear resistance of chromium-molybdenum coatings.
- Wear specimens plated at 43°C have larger grain structures, higher molybdenum contents, and longer wear life than those plated at room temperature.
- Pulse-plating of chromium does not improve its wear resistance. Optimum temperature for chromium plating is 49°C.
- A pulse setting of 3 ms on-time, 3 ms off-time maximizes the wear resistance of chromium-molybdenum on titanium.
- The optimum diffusion heat-treat cycle for Ti 6Al-4V is 760°C for 3 hr followed by 538°C for 6 hr.
- The optimum diffusion heat-treat cycle for Ti 8Al-1Mo-1V is 954°C for 1 hr followed by 593°C for 8 hr.

- The use of a dry-film lubricant substantially improves the wear life of chromium-molybdenum and chromium.

#### **CORROSION RESISTANCE**

- The corrosion resistance of chromium-molybdenum is optimized by plating at a pulse setting of 50 ms on-time, 3 ms off-time.

#### **LOW-CYCLE FATIGUE**

- The chromium-molybdenum plating/diffusion process is detrimental to the low-cycle fatigue life of Ti 8Al-1Mo-1V, but the oxidation resistance afforded by the coating gradually compensates for this effect as temperature is increased.

#### **COMPONENT TESTING**

- The use of electroplated chromium-molybdenum and a dry-film lubricant significantly increased the galling resistance of a titanium fan blade root compared to a dry-film lubricant alone, but results were little better than those for an uncoated root.

#### **SECTION IV RECOMMENDATIONS**

Based on the results of the first three phases of this study, the following recommendations are made for continued development and evaluation of chromium-molybdenum coatings. This continuation will provide the next step in obtaining sufficient data on the coating system to allow wide use of chromium-molybdenum on aerospace system components.

- Conduct a more intensive evaluation of the influence of dry-film lubricants on the wear resistance of chromium-molybdenum coatings
- Apply the coating to a variety of components for functional testing
- Provide detailed procedures for the application of chromium-molybdenum coatings to meet the requirements of the proposed Military Specification shown as Appendix D
- Provide specific inspection/control methods, such as coating thickness determination, plating solution analysis, and surface finish control.



## APPENDIX A

### CHROMIUM-MOLYBDENUM APPLICATION PROCEDURE FOR TITANIUM

#### SOLUTION COMPOSITION:

##### Self-Regulating Plating Bath

|   |                     |
|---|---------------------|
| Unichrome <sup>(1)</sup>  | 300 g/l (40 oz/gal) |
| Ammonium Molybdate<br>(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> · 4H <sub>2</sub> O | 75 g/l (10 oz/gal)  |

1. Dissolve the chromic acid in deionized water at room temperature
2. Dissolve the ammonium molybdate in deionized water at 60 ± 5°C (140 ± 10°F)
3. Add the ammonium molybdate solution to the chromic acid and adjust the volume to operating level.

##### Vapor Blast

|   |                                |
|---|--------------------------------|
| Novaculite 200 <sup>(2)</sup>           | 480 to 720 g/l (4 to 6 lb/gal) |
| Antisolidifying Compound <sup>(3)</sup> | 113 g/22.7 kg (4 oz/50 lb)     |
| Corrosion Inhibitor <sup>(4)</sup>      | 4 ml/l (15 ml/gal)             |

1. Fill tank to about ¾ of operating level with water
2. Add abrasive slowly while circulating pump is on
3. Add antisolidifying compound
4. Add corrosion inhibitor
5. Fill to operating level.

##### Etch Solution

|                         |                     |
|-------------------------|---------------------|
| Hydrofluoric Acid (48%) | 2.1 ml/l (8 ml/gal) |
| Sodium Chromate         | 20 g/l              |

1. Dissolve the required amount of sodium chromate in deionized water
2. Add required amount of hydrofluoric acid.

#### PROCEDURE FOR PLATING:

1. Mask area not to be plated
2. Vapor blast surface to be plated with wet abrasive at 0.4 to 0.7 MPa (50 to 100 psi)

(1)M&T Chemicals, Inc., SRHS CR 110

(2)Vapor Blasting Manufacturing Co., NVB No. 200

(3)Vapor Blasting Manufacturing Co., No-Pak

(4)Reilly-Whiteman-Walton Co., X61-60 Water Conditioner.

3. Rinse thoroughly in clean running water. Cleaned surface must not be allowed to dry prior to plating. Submerge part in deionized water
4. Etch for 5 to 10 sec to produce chrome conversion coating
5. Using lead anodes, immerse part in plating solution with current "off"
6. Turn on power supply and raise current to 11.7 ASD (0.75 ASI) for 2.5 min
7. Plate at 46.7 ASD (3.0 ASI) for 30 min with pulse plater set at desired "on"/"off" cycle. Setting off-time to zero results in direct current plating
8. Rinse thoroughly in deionized water
9. Heat-treat in vacuum as follows:

|                |                          |
|----------------|--------------------------|
| Ti 8Al-1Mo-1V: | 954°C (1750°F) for 1 hr  |
| followed by    | 593°C (1100°F) for 8 hr  |
| Ti 6Al-4V:     | 760°C (1400°F) for 3 hr  |
| followed by    | 538°C (1000°F) for 6 hr. |

For other titanium alloys, the diffusion heat-treat cycle should be compatible with previous heat-treat history and selected in accordance with MIL-H-81200.

10. (Option) — Vapor blast surfaces.

## APPENDIX B

### CHROMIUM-MOLYBDENUM APPLICATION PROCEDURE FOR STEELS

#### SOLUTION COMPOSITION:

##### Self-Regulating Plating Bath

|   |                     |
|---|---------------------|
| Unichrome <sup>(1)</sup>  | 300 g/l (40 oz/gal) |
| Ammonium Molybdate<br>(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> · 4H <sub>2</sub> O | 75 g/l (10 oz/gal)  |

1. Dissolve the chromic acid in deionized water at room temperature
2. Dissolve the ammonium molybdate in deionized water at 60 ± 5°C (140 ± 10°F)
3. Add the ammonium molybdate solution to the chromic acid and adjust the volume to operating level.

#### STRESS RELIEF TREATMENT

All steel parts having an ultimate tensile strength of 1,034 MPa (150,000 psi) and above, which are machined, ground, cold-formed or cold-straightened, shall be baked at a minimum of 191 ± 14°C (375 ± 25°F) for 3 hr or more prior to cleaning and plating for the relief of damaging residual tensile stress.

#### PROCEDURE FOR PLATING:

1. Mask area not to be plated
2. Anodic etch at 1 to 3 amps/in.<sup>2</sup> for 0.5 to 10 min. Etched in the following solution (preferred) or in the self-regulating plating bath described above:

|              |                                  |
|--------------|----------------------------------|
| Chromic Acid | 225 to 300 g/l (30 to 40 oz/gal) |
| Temperature  | 32 to 54°C (90 to 130°F)         |

Note: Etching increases iron contamination in the plating bath; therefore, the use of this solution is recommended over the plating bath.

3. Using lead anodes, immerse part in plating solution with current "off"
4. Turn on power supply and raise current to 11.7 ASD (0.75 ASI) for 2.5 min
5. Plate at 46.7 ASD (3.0 ASI) for 30 min with pulse plater set at desired "on"/"off" cycle. Setting off-time to zero results in direct current plating

6. Rinse thoroughly in clear running water

7. Embrittlement Relief

All coated steel parts having a hardness of Rockwell C40 and higher shall be baked at a minimum of  $191 \pm 14^{\circ}\text{C}$  ( $375 \pm 25^{\circ}\text{F}$ ) for 3 hr or more, within 4 hr after plating, to provide hydrogen-embrittlement relief

8. (Option) — Vapor blast surfaces.

## APPENDIX C

### CHROMIUM TRIOXIDE PLATING BATH

#### SOLUTION COMPOSITION

|  |                     |
|--|---------------------|
| Chromium Trioxide<br>$\text{CrO}_3$  | 300 g/l (40 oz/gal) |
| Ammonium Molybdate<br>$(\text{NH}_4)_6 \text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ | 75 g/l (10 oz/gal)  |

1. Dissolve the chromium trioxide in deionized water at room temperature
2. Dissolve the ammonium molybdate in deionized water at  $60 \pm 5^\circ\text{C}$  ( $140 \pm 10^\circ\text{F}$ )
3. Add the ammonium molybdate solution to the chromic acid and adjust volume to operating level
4. Sulfuric acid must be added to the solution as a catalyst.

**APPENDIX D**  
**SUGGESTED**  
**MILITARY SPECIFICATION**

**CHROMIUM-MOLYBDENUM PLATING**  
**(ELECTRODEPOSITED)**

**1. SCOPE**

**1.1 Scope.** This specification covers the requirements for electrodeposited chromium-molybdenum plating on steel and titanium alloys.

**2. APPLICABLE DOCUMENTS**

**2.1** The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

*Military Specifications:*

|             |   |
|-------------|---|
| MIL-S-5002  | Surface Treatments and Inorganic Coatings for Metal Surfaces of Weapons Systems |
| MIL-S-13165 | Shot Peening of Ferrous Parts   |
| MIL-H-81200 | Heat Treatment of Titanium and Titanium Alloys                                  |
| MIL-R-81841 | Rotary Flap Peening of Metal Parts  |

*Military Standards:*

|              |   |
|--------------|---|
| MIL-STD-105  | Sampling Procedures and Tables for Inspection by Attributes |
| MIL-STD-1312 | Fasteners, Test Methods                                     |

(Copies of Military Specifications and Standards required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

*Federal Specifications:*

|          |  |
|----------|--|
| QQ-S-624 | Steel Bar, Alloy, Hot Rolled and Cold Finished (General Purpose) |
|----------|--|

(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards and Handbooks as outlined under General Information in the Index of Federal Specifications and Standards and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.)

(Single copies of this specification and other Federal Specifications required by activities outside the Federal Government for bidding purposes are available without charge from Business Service Centers at the General Services Administration Regional Offices in Boston, New York, Washington, DC, Atlanta, Chicago, Kansas City, MO, Fort Worth, Denver, San Francisco, Los Angeles, and Seattle.)

(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

**2.2 Other Publications.** The following documents form a part of this specification to the extent specified herein. Unless a specific issue is identified, the issue in effect on date of invitation for bids or request for proposal shall apply.

*American Society for Testing and Materials (ASTM) Standards:*

|            |   |
|------------|---|
| ASTM B-481 | Preparation of Titanium and Titanium Alloys for Electroplating  |
| ASTM B-487 | Measurement of Metal and Oxide Coating Thickness by Microscopic Examination of a Cross Section          |
| ASTM B-499 | Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metal |
| ASTM B-504 | Measuring the Thickness of Metallic Coatings by the Coulometric Method                                  |
| ASTM B-578 | Measurement of Microhardness of Electroplated Coatings  |
| ASTM E-8   | Tension Testing of Metallic Materials   |

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

### **3. REQUIREMENTS**

**3.1 Materials and Operating Conditions.** The materials used shall be such as to produce platings which meet the requirements of this specification. The deposition of chromium-molybdenum shall be out of a chromic acid type bath supplemented with a molybdate salt. Bath composition and operating conditions shall be such as to produce a satisfactory bond between the basis metal and the coating.

#### **3.2 General Requirements.**

**3.2.1 High Tensile Steel Parts.** Unless otherwise specified (see 6.2), steel parts having an ultimate tensile strength greater than 1655 MPa (240,000 psi) shall not be plated without specific approval of the procuring activity.

**3.2.2 Stress Relief Treatment.** All steel parts having an ultimate tensile strength of 1034 MPa (150,000 psi) and above, which are machined, ground, cold formed, or cold straightened, shall be baked at a minimum of  $191 \pm 14^{\circ}\text{C}$  ( $375 \pm 25^{\circ}\text{F}$ ) for three hours or more prior to cleaning and plating for the relief of damaging residual tensile stresses. When peening is required (see 3.3.3 and 3.3.5), thermal stress relief shall be performed prior to shot or rotary flap peening. Stress relief of titanium parts shall be in accordance with MIL-H-81200.

**3.2.3 Surface Preparation.**

**3.2.3.1 Cleaning.** Unless otherwise specified (see 6.2), all steel parts shall be cleaned in accordance with MIL-S-5002. Other base metals shall be cleaned by methods which shall not damage the substrate and shall not interfere with adhesion of the deposit. Vapor blasting is preferred for cleaning titanium parts.

**3.2.3.2 Activating.** Unless otherwise specified (see 6.2), all steel parts shall be activated by electrolytic etching in a chromic acid type bath. Titanium parts shall be activated in accordance with ASTM B-481, Process No. 1, omitting the pickling step, 2.2.1.2.

**3.2.4 Plating Application.** Unless otherwise specified (see 6.2), the plating shall be applied after all base metal heat treatments and mechanical operations, such as machining, brazing, welding, forming, and perforating of the article, have been completed.

**3.2.5 Embrittlement Relief.** All coated steel parts having a hardness of Rockwell C40 and higher shall be baked at a minimum of  $191 \pm 14^{\circ}\text{C}$  ( $375 \pm 25^{\circ}\text{F}$ ) for three hours or more, within four hours after plating, to provide hydrogen embrittlement relief (see 6.4). The baked parts, when tested in accordance with 4.5.5, shall not crack or fail by fracture (see 4.4.3.5). Plated springs and other parts subject to flexure shall not be flexed prior to hydrogen embrittlement relief treatment.

**3.2.6 Diffusion Heat-Treat Cycle.** All coated titanium parts should be heat treated to facilitate adhesion of the deposit and to provide hydrogen embrittlement relief. This diffusion cycle should be compatible with the previous heat-treat history of the base metal and selected in accordance with MIL-H-81200 unless otherwise specified (see 6.2). Titanium alloys not covered by MIL-H-81200 should be vacuum heat treated at  $760^{\circ}\text{C}$  ( $1400^{\circ}\text{F}$ ) for 3 hours.

**3.2.7 Coverage.** Unless otherwise specified (see 6.2), the plating shall cover all surfaces including roots of threads, corners, and recesses.

**3.2.8 Boundaries.** Boundaries of chromium-molybdenum plating which covers only a portion of the surface shall be free from beads, nodules, jagged edges, and other irregularities.

**3.2.9 Finish.** A gray matte finish, relatively smooth and free from blackened or frosty areas, shall be acceptable. Shading will vary depending on the molybdenum content of the deposit.

**3.3 Processing of Steel Parts.** Unless otherwise specified (see 6.2), steel parts shall be processed in accordance with the procedural instructions of the procuring activity as follows:



**3.3.1 Class 1.** Parts shall be plated or plated and processed to specific dimensions in accordance with procedures and criteria specified by the procuring activity. Parts not covered by procedural instructions which do not specify baking procedures shall be baked in accordance with 3.2.5 after plating.

**3.3.2 Class 2.** Plated parts below Rockwell C40 hardness, which are subjected to static loads or designed for limited life under dynamic loads or combinations thereof, need not be peened prior to plating.

**3.3.3 Class 3.** Plated parts below Rockwell C40 hardness, which are designed for unlimited life under dynamic loads, shall be peened in accordance with MIL-S-13165 or MIL-R-81841 prior to plating. Unless otherwise specified in the applicable drawings, the peening shall be accomplished on all surfaces for which the plating is required and on all immediately adjacent surfaces when they contain notches, fillets, or other abrupt changes in section size where stress will be concentrated.

**3.3.4 Class 4.** Plated parts, Rockwell C40 hardness or above, which are subjected to static loads or designed for limited life under dynamic loads or combinations thereof, shall be baked in accordance with 3.2.5 after plating. The load for the static load test (see 4.5.5, the embrittlement relief test) shall be as specified in the contract, order, or applicable drawing (see 6.2).

**3.3.5 Class 5.** Plated parts, Rockwell C40 hardness or above, which are designed for unlimited life under dynamic loads, shall be peened in accordance with MIL-S-13165 or MIL-R-81841 prior to plating. Unless otherwise specified in the applicable drawings, the peening shall be accomplished on all surfaces for which the plating is required and on all immediately adjacent surfaces when they contain notches, fillets, or other abrupt changes in section size where stress will be concentrated. The plated parts shall be baked in accordance with 3.2.5 after plating. The load for the static load test (see 4.5.5, the embrittlement relief test) and the dynamic load conditions shall be as specified in the contract, order, or applicable drawing (see 6.2).

#### **3.4 Detail Requirements.**

**3.4.1 Thickness.** The minimum, maximum, or range of thickness for chromium-molybdenum plating shall be as specified in the contract, purchase order, or on the applicable drawing (see 6.2). If a thickness is not specified, the minimum thickness for the finished part shall be 0.0005 in., or 0.5 mil (0.013 mm). The thickness requirement shall apply after all metal finishing and post-plating grinding operations have been completed.

**3.4.2 Adhesion.** The adhesion of the plating and any underplate shall be such that when examined at a magnification of approximately 4 diameters, neither the plating, nor any electro-deposited underplate, shall show separation from the base metal or from each other at their common interface(s) when subjected to the test described in 4.5.2. The interface between a plating and the base metal is the surface of the base metal before plating. The formation of cracks in the base metal or the plate which do not result in flaking, peeling, or blistering of the plate shall not be cause for rejection.

**3.4.3 Hardness.** The minimum hardness of a cross-section of the plating, when subjected to the microhardness test detailed in 4.5.3, shall be 200 Vickers Hardness Number (VHN).

**3.4.4 Porosity.** The chromium-molybdenum plating, by being as free from porosity as possible, shall be capable of protecting steel parts from corrosion due to pits, pores, or cracking. When subjected to the test detailed in 4.5.4, specimens shall show no more than a total of 15 isolated spots or pits, none larger than 0.79 mm (0.03 in.) in diameter, in a total of 967.8 sq cm (150 sq in.) of test area grouped from five or more test pieces; nor more than five isolated spots or pits, none larger than 0.79 mm (0.03 in.) in a total of 193.6 sq cm (30 sq in.) from one or more test pieces, except those areas within 1.59 mm (0.06 in.) from identification markings and contact marks after processing.

### **3.5 Workmanship.**

**3.5.1 Base Metal.** The base metal shall be free from visible defects that will be detrimental to the appearance or protective value of the plating. The base metal shall be subjected to such cleaning and plating procedures as necessary to yield deposits herein specified.

**3.5.2 Plating.** The plating shall be smooth, fine grained, adherent, uniform in appearance, free from blisters, pits, nodules, excessive edge build-up, and other defects. The plating shall show no indication of contamination or improper operation of equipment used to produce the deposit, such as excessively powdered or darkened plating, build-up, and other defects. The size and number of contact marks shall be at a minimum consistent with good practice. The location of contact marks shall be in areas of minimum exposure to service environmental conditions where important to the function of the part. Superficial staining which has been demonstrated as resulting from rinsing or slight discoloration resulting from baking operations to relieve embrittlement, as specified above (see 3.2.5), shall not be cause for rejection. All details of workmanship shall conform to the best practice for high quality plating.

## **4. QUALITY ASSURANCE PROVISIONS**

**4.1 Responsibility for Inspection.** Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

**4.2 Classification of Inspection.** The inspection requirements specified herein are classified as follows:

- a. Production Control Inspection (see 4.3)
- b. Quality Conformance Inspection (see 4.4).

### **4.3 Production Control Inspection.**

**4.3.1 Control Records.** When specified in the contract or order (see 6.2), the supplier shall maintain a record of each processing bath, showing all additional chemicals or treatment solutions to the unit, the results of all chemical analyses performed, and the quantity of parts plated during operation. Upon request of the procuring activity, such records, as well as reports of the test results, shall be made available. These records shall be maintained for not less than one year after completion of the contract or purchase order.

**4.3.2 Production Control.** The equipment, procedures, and operations employed by a supplier shall be capable of producing high quality electrodeposited platings as specified in this document. When specified by the procuring activity (see 6.2), the supplier, prior to production, shall demonstrate the capability of the process used to show freedom from hydrogen embrittlement damage as indicated by satisfactory behavior of specimens prepared (see 6.2.2) and tested in accordance with 4.3.2.1 to comply to the requirements of MIL-S-5002 for preproduction process qualifications.

**4.3.2.1 Preproduction Control.** For preproduction control, four round notched steel specimens shall be prepared in accordance with 4.4.4.3 from four individual heats for a total of 16 specimens, using the specified steel alloy for which preproduction examinations of the process is to be demonstrated. Specimens shall be heat treated to the maximum tensile strength representing production usage. The specimens shall be given the same pretreatments and treatments proposed for production. The specimens shall be subject to the test detailed in 4.5.5. The process shall be considered satisfactory if all specimens show no indication of cracks or failure. The test results and production control information shall be submitted to the procuring activity for approval. Until approval has been received parts shall not be plated.

**4.3.3 Frequency of Tests.** To assure continuous control of the process as required by MIL-S-5002 and to prevent detrimental hydrogen embrittlement during production, the satisfactory behavior of specimens prepared and tested in accordance with Table I shall be made once each month or more frequently if required by the procuring activity. The results of tests made to determine conformation of electrodeposited platings to all requirements of this specification are acceptable as evidence of the properties being obtained with the equipment and procedures employed.

TABLE I. PRODUCTION CONTROL TESTS AND SPECIMENS

| Test                          | Requirement Paragraphs | Specimen Preparation Paragraph <sup>(1)</sup> | Test Reference Paragraphs |
|-------------------------------|------------------------|---|---------------------------|
| Thickness                     | 3.4.1                  | 4.4.4 and 4.4.4.1                             | 4.5.1                     |
| Adhesion                      | 3.4.2                  | 4.4.4 and 4.4.4.1                             | 4.5.2                     |
| Hardness                      | 3.4.3                  | 4.4.4 and 4.4.4.1                             | 4.5.3                     |
| Porosity                      | 3.4.4                  | 4.4.4 and 4.4.4.2                             | 4.5.4                     |
| Hydrogen Embrittlement Relief | 3.2.5                  | 4.4.4 and 4.4.4.3                             | 4.5.5                     |

<sup>(1)</sup> Standard alloy steels shall be used for production control specimens. The selection shall be at the option of the supplier; however, alloy steels such as AISI or SAE numbers 4130, 4135, 4140, 4145, 4340, 8645 and 8740 conforming to QQ-S-624 shall be used.

**4.3.4 Production Control Specimens.** Test specimens for production control shall be prepared in accordance with 4.4.4, 4.4.4.1 and 4.4.4.2, as applicable for the thickness, adhesion, hardness, and porosity tests detailed in Table I. specimens for the production control embrittlement relief test shall be four round notched steel specimens of alloy steel 4340, conforming to QQ-S-624, heat treated to the maximum tensile strength from one or more heats, and prepared in accordance with 4.4.4.3.

#### 4.4 Quality Conformance Inspection.

**4.4.1 Lot.** A lot shall consist of plated articles of the same base metal composition, class, deposition form and finish, plated and treated under the same conditions, and approximately the same size and shape submitted for inspection at one time.

**4.4.2 Sampling for Visual Examination and Nondestructive Tests.** Sampling for visual examination and nondestructive tests shall be conducted as directed by the procuring activity (see 6.2) in accordance with MIL-STD-105 or using Table II. A sample of coated parts or articles shall be drawn by taking at random from each lot the number of articles in accordance with MIL-STD-105, Level II, Acceptable Quality Level (AQL) 1.5 percent defective or as indicated in Table II. The lot shall be accepted or rejected according to the procedures in 4.4.2.1 for visual examination and 4.4.2.2 for plating thickness (nondestructive tests).

TABLE II. SAMPLING FOR VISUAL EXAMINATION AND NONDESTRUCTIVE TESTS

| <i>Number of<br/>Items in<br/>Lot Inspections</i> | <i>Number of Items<br/>in Samples<br/>(Randomly Selected)</i> | <i>Acceptance Number<br/>(maximum number of<br/>sample items nonconforming<br/>to any test)</i> |
|---|---|---|
| 15 or less  | 7 <sup>1</sup>  | 0   |
| 16 to 40  | 10  | 0   |
| 41 to 110   | 15  | 0   |
| 111 to 300  | 25  | 1   |
| 301 to 500  | 35  | 1   |
| 501 and over                                      | 50  | 2   |

<sup>1</sup> If the number of items in the inspection lot is less than 7, the number of items in the sample shall equal the number of items in the inspection lot.

**4.4.2.1 Visual Examination.** Samples selected in accordance with 4.4.2 shall be examined for compliance with the requirements of 3.2.7, 3.2.8, 3.2.9, and 3.5.2 after plating. If the number of nonconforming articles exceeds the acceptance number for the sample, the lot represented by the sample shall be rejected.

**4.4.2.2 Thickness of Plating (Nondestructive Tests).** Samples selected in accordance with 4.4.2 shall be inspected and the plating thickness measured by the applicable test detailed in 4.5.1 at several locations on each article as defined in 3.4.1 for compliance with the requirement. Measurements on fastener hardware shall be made on locations defined in MIL-STD-1312, Test 12. The part or article shall be considered nonconforming if one or more measurements fail to meet the specified minimum thickness. If the number of defective items in any sample exceeds the acceptance number for the specified sample, the lot represented by the sample shall be rejected. Separate specimens (see 4.4.4.1) shall not be used for thickness measurements unless a need has been demonstrated.

**4.4.3 Sampling for Destructive Tests.** A random sample of five plated parts or articles shall be taken from each lot for each destructive test or separately plated specimens shall be prepared in accordance with 4.4.4, 4.4.4.1, 4.4.4.2, and 4.4.4.3 to represent each lot. If the number of articles in the lot is five or less, the number of articles in the sample shall be specified by the procuring activity (see 6.2).

**4.4.3.1 Thickness of Plating (Destructive Tests).** If sampling and testing for the thickness of plating by nondestructive testing is not the option of the supplier, samples selected in accordance with 4.4.3 shall be measured for plating thickness by the applicable tests detailed in 4.5.1 at several locations as defined in 3.4.1 for compliance with the requirements. Measurements for fastener hardware shall be made at locations defined in MIL-STD-1312, Test 12. If the plating thickness at any place on any article or specimen is less than the specified minimum thickness, the lot shall be rejected. Separate specimens (see 4.4.4.1) shall not be used for thickness measurements unless a need has been demonstrated.

**4.4.3.2 Adhesion (Destructive Tests).** The articles or specimens used for the destructive thickness test (see 4.4.3.1), if of suitable size and form, may be used as the test pieces for the adhesion test to determine compliance with the requirements of 3.4.2. Failure of one or more of the test pieces shall constitute failure of the lot.

**4.4.3.3 Hardness (Destructive Tests).** When specified in the contract or offer (see 6.2), compliance with the requirements for hardness shall be determined. The articles or specimen used for the destructive thickness test (see 4.4.3.1), if of suitable size and form, may be used as the test pieces for examination to determine compliance with the requirement of 3.4.3. Failure of one or more of the test pieces shall constitute failure of the lot.

**4.4.3.4 Porosity (Destructive Tests).** When specified in the contract or order (see 6.2), compliance with the requirements for porosity of chromium-molybdenum on steel shall be determined. A set of five separate test specimens prepared in accordance with 4.4.4 and 4.4.4.2 in lieu of treated plated articles shall be used to determine compliance with the requirements for porosity (see 3.4.4). Failure of one or more of the test specimens shall reject the lot.

**4.4.3.5 Hydrogen Embrittlement Relief (Destructive Tests).** Unless otherwise specified in the contract or offer (see 6.2), conformance to the requirements of 3.2.5 for hydrogen embrittlement relief of treated steel parts shall be determined for those parts, comprising a lot, having a tensile strength of or heat treated to a tensile strength level of 1655 MPa (240,000 psi) or above and which will be subjected to a sustained tensile load in use. A random sample of five plated articles shall be taken from each lot or five specimens, prepared in accordance with 4.4.4 and 4.4.4.3, shall be used to represent the lot. When tested as specified in 4.5.5, cracks or failure by fracture shall be cause for rejection. Failure of one or more of the test pieces shall reject the lot.

**4.4.4 Quality Conformance Specimen Preparation.** When the plated articles are of such form, shape, size, and value as to prohibit use thereof, or are not readily adaptable to a test specified herein, or when destructive tests of small lot sizes are required, the test shall be made by the use of separate specimens plated concurrently with the articles represented. The separate specimens shall be of a base metal equivalent to that of the article represented. "Equivalent" base metal includes chemical composition, grade, condition and finish of surface prior to plating. For example, a cold-rolled steel surface should not be used to represent a hot-rolled steel surface. Due to the impracticality of forging or casting separate test specimens, hot-rolled specimens may be used to represent forged and cast-steel articles. The separate specimens may also be cut from the scrap casting when ferrous alloy castings are being plated. These separate specimens may be introduced into a lot at regular intervals prior to the cleaning operations, prior to plating, and shall not be separated therefrom until after completion of plating. Conditions affecting the plating of specimens, including the spacing, plating media, bath agitation, temperature, etc., in respect to other objects being plated shall correspond as nearly as possible to those affecting the significant surfaces of the articles represented. Separate specimens shall not be used for thickness measurements, however, unless the necessity for their use has been demonstrated.

**4.4.4.1 Specimens for Thickness, Adhesion, and Hardness Tests.** If separate specimens for thickness, adhesion, and hardness tests are required, they shall be strips approximately 25 mm (1 in.) wide, 102 mm (4 in.) long and 1 mm (0.04 in.) thick.

**4.4.4.2 Specimens for Porosity Tests.** If separate specimens for porosity tests are required, they shall be panels not less than 254 mm (10 in.) in length, 76 mm (3 in.) in width and approximately 1 mm (0.04 in.) thick.

**4.4.4.3 Specimens for Embrittlement Relief.** Separate specimens for embrittlement relief test shall be round notched specimens with the axis of the specimen (load direction) perpendicular to the short traverse grain flow direction. The configuration shall be in accordance with Figure 8 of ASTM E-8 for rounded specimens. Specimens shall have a 60 deg V-notch located approximately at the center of the gage length. The cross-section area at the root of the vee shall be approximately equal to half the area of the full cross-section area of the specimen's reduced section. The vee shall have a  $0.254 \pm 0.0127$  mm ( $0.010 \pm 0.0005$  in.) radius of curvature at the base of the notch (see 6.2.2).

## **4.5 Tests.**

**4.5.1 Thickness.** For nondestructive measurement of plating thickness on steel, procedures in accordance with ASTM B-499 (Magnetic Test Method) may be used. On titanium alloys, plating thickness may be measured with an eddy current-type gage. For destructive measurement of plating thickness, procedures in accordance with ASTM B-487 (Microscopic) or ASTM B-504 (Coulometric) may be used. In addition to the above, other procedures embodied in MIL-STD-1312, Test 12, may be used for thickness measurement of plated fastener hardware.

**4.5.2 Adhesion.** Adhesion may be determined by scooping the surface or shearing with a sharp edge, knife, or razor through the plating down to the base metal and examining at four diameters magnification for evidence of non-adhesion. Alternately, the article or specimen may be clamped in a vise and the projecting portion bent back and forth until rupture occurs. If the edge of the ruptured plating can be peeled back or if separation between the plating and the base metal can be seen at the point of rupture when examined at four diameters magnification, adhesion is not satisfactory.

**4.5.3 Hardness.** The hardness of plating shall be determined by a microhardness traverse in accordance with ASTM B-578, except that a Vicker indenter and 100 gram load shall be used. A minimum of three hardness readings shall be made to establish the base metal hardness in an area at least 3.175 mm (0.125 in.) from the outer surface or at mid radius of the cross section whichever is less. Readings shall be taken at 0.013 mm (0.0005 in.) intervals starting at 0.025 mm (0.001 in.) from the outer surface in a staggered pattern until the pre-established base metal hardness is reached. The hardness reading may be plotted versus distance from the outer surface. The point at which the hardness shows a vast decrease may be taken as the limits of chromium-molybdenum plating.

**4.5.4 Porosity.** Prior to determining porosity of chromium-molybdenum on steel by the ferroxyl test, the specimen surface shall be cleaned to remove any oil or grease. Contamination removal shall be accomplished with any acceptable solvent in accordance with MIL-S-5002. A sheet of filter paper, saturated by dipping in a ferroxyl solution heated to 82 to 94°C (180 to 200°F), shall be applied to the flat surface of the specimen or of the article. The solution composition shall be as follows:

|  |                 |
|--|-----------------|
| Potassium ferricyanide ( $K_3Fe(CN)_6$ ) | 1 gm            |
| Sodium chloride (NaCl)                   | 10 gm           |
| Agar                                     | 10 gm           |
| Water (distilled or deionized)           | to make 1 litre |

After 10 min, the heated filter paper shall be removed. Both the plated surface and the filter paper shall be examined. Where corrosion of the base metal will occur at pores or other defects due to the plating, dark blue spots will have been developed. Contact may further be assured by the use of a soft bristle brush moistened with the reagent solution. For a permanent record, the filter paper can be dried.

**4.5.5 Embrittlement Relief.** Compliance with 3.2.5 shall be determined with samples of plated parts as specified in 4.4.3.5. Parts such as spring pins, lock rings, etc., which are installed in holes or rods, shall be similarly assembled using the applicable parts specifications or drawings tolerances which impose the maximum sustained tensile load on the plated part. The selected samples shall be subjected to a sustained tensile load equal to 115 percent of the maximum design yield load for which the part was designed. Parts which require special fixtures, extreme loads to comply with the above requirements, or where the maximum design yield load is not known, may be represented by separate specimens prepared in accordance with 4.4.4.3. The notched samples shall be subjected to a sustained tensile load equal to 75 percent of the ultimate notch tensile strength of the material. The articles, parts or specimens shall be held under load for at least 200 hours and then examined for cracks or fracture.

## **5. PREPARATION FOR DELIVERY**

**5.1 Packaging and Packing.** Preservation, packaging, and packing methods for electro-deposited plated parts or articles employed by a supplier shall be such as to preclude damaging during shipment and handling.

## **6. NOTES**

**6.1 Intended Use.** Chromium-molybdenum plating is used for wear resistance, abrasion resistance, and such incidental corrosion protection of parts as the specified thickness of the plating may afford. Heavy deposits of the plating may be used for buildup of worn or undersized parts, or for salvage purposes, and to provide protection against corrosive chemical environments.

**6.2 Ordering Data.** Purchasers should select the preferred options permitted herein and include the following information in procurement documents.

- a. Title, number, and date of this specification
- b. Class of plating (see 1.2, 3.3, 3.3.1, 3.3.2, 3.3.3, 3.3.4, and 3.3.5)
- c. When plating is to be applied, if other than specified (see 3.2.1, 3.2.4, 3.3, 3.3.1, 3.3.2, 3.3.3, 3.3.4, and 3.3.5)
- d. Surface preparation, if other than specified (see 3.2.3)
- e. Heat-treat cycle, if other than specified (see 3.2.6)
- f. Coverage, if other than specified (see 3.2.7)
- g. Surface finish, if particular finish required (see 3.2.9)
- h. Thickness of plating, as specified (see 3.3, 3.3.1, and 3.4.1)

- i. Control record requirement (see 4.3.1)
- j. Preproduction control examination (see 4.3.2)
- k. Sampling plan (see 4.4.2)
- l. Number of samples for destructive testing (see 4.4.3)
- m. Hardness, porosity, and hydrogen embrittlement tests, whether required for quality conformance inspection (see 4.4.3.3, 4.4.3.4, and 4.4.3.5).

**6.2.1** The manufacturer of the base metal parts should provide the plating facility with the following data:

- a. Hardness of steel parts (see 3.2.1, 3.2.2, 3.2.5, and 3.3)
- b. Heat treatment for stress relief, whether has been performed or is required (see 3.2.2)
- c. Tensile loads required for embrittlement relief test, if applicable (see 3.2.5 and 4.5.5).

**6.2.2** The manufacturer of the base metal parts should provide the plating facility with notched specimens (see 4.4.4.3) to be plated for conformance with 3.2.5 required for production control (see 4.3.2.1 and 4.3.4) and for lot acceptance (see 4.4.3 and 4.4.3.5).

**6.3 Stress Relief.** There is a hazard that hardened and tempered cold-worked or cold-straightened steel parts may crack during cleaning and plating. Such parts shall have a suitable heat treatment for stress relief prior to cleaning and plating (see 3.2.2).

**6.4 Baking Time.** For high strength steels (Rockwell C40 and above), it may be beneficial to extend the baking time to 23 hours to ensure complete hydrogen embrittlement relief (see 3.2.5).



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